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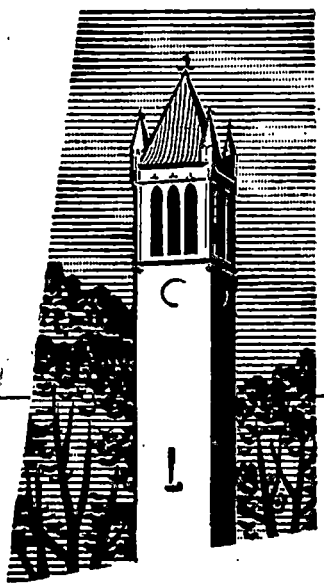
METHODS FOR TESTING ENGINEERING SOILS

by
D. T. Davidson
and
Associates



IOWA ENGINEERING EXPERIMENT STATION

Iowa State University
Ames, Iowa

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CONTENTS

PREFACE

This bulletin is a further compilation of the reports on completed research done for the Iowa State Highway Research Board Project HR-1 **The loess and glacial till materials of Iowa; an investigation of their physical and chemical properties and techniques for processing them to increase their all-weather stability for road construction.** The research, started in 1950, was done by the Iowa Engineering Experiment Station at Iowa State University under its project 283-S. The project was supported by funds from the Iowa State Highway Commission.

The principal objectives of the project may be summed up as follows:

1. To determine by means of both field and laboratory studies the areal and stratigraphic variation in the physical and chemical properties of the loess and glacial till materials of Iowa.
2. To develop new equipment and methods for evaluating physical and chemical properties of soil where needed.
3. To correlate fundamental soil properties with the performance of soils in the highway structure.
4. To develop a scientific approach to the problem of soil stabilization based on the relationships between the properties of the soils and those of the admixtures.
5. To determine the manner in which the loess and glacial till materials of Iowa can be processed for optimum performance as highway embankments, sub-grades, base courses, and surface courses.

Many of the papers in this bulletin were prepared originally as graduate theses required for master or doctoral degrees. Each was then rewritten with the assistance of other project workers and was submitted to the Iowa Highway Research Board as a report on a phase of completed research. This explains the several authors for each paper. The research work was all done under Dr. D. T. Davidson as project leader in charge.

Practically all the papers herein have been published previously. The title page for each manuscript identifies all authors and gives the place and date of first publication. No attempt has been made to revise, update, and change the data; hence some contradictions are evident. The facts and conclusions presented are those of the authors at the time the manuscript was submitted. Much of the repetition of material has been eliminated, and the papers have been arranged by subject matter.

The list of REFERENCES at the end of each manuscript gives only the first, or original printing, though the paper referred to may have appeared later in various forms in several publications, and some are included herein. Those shown as theses in the Iowa State University Library are so indicated, because only in the theses are all the data shown.

DISPERSION OF LOESS FOR MECHANICAL ANALYSIS

by

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(Highway Research Board Proceedings, 31:500-510. 1952.)

ABSTRACT

A special study was made in connection with the investigation of the Wisconsin (Peorian) loess of southwestern Iowa, which is being carried on jointly by the Engineering Experiment Station of Iowa State University and the Iowa State Highway Commission to develop a tailored procedure for dispersing Wisconsin loess for mechanical analysis. The principle of dispersion is reviewed to clarify the function of each step in procedures and also to serve as a guide for the development of a dispersion procedure for Wisconsin loess.

In addition, experiments were performed to: (1) evaluate several deflocculating agents, sodium metasphosphate, sodium pyrophosphate, sodium oxalate, and sodium silicate; (2) determine the need for hydrogen-peroxide and hydro-chloric-acid treatments; and (3) evaluate a rubber stirring paddle and determine stirring time requirements, using the conventional high-speed malted milk type mixing apparatus.

Among the deflocculating agents compared experimentally, sodium metaphosphate is the most efficient in dispersing Wisconsin loess. The amount of this chemical adopted for dispersing Wisconsin loess samples for mechanical analysis determinations is 60 ml. of 0.5 N solution.

Wisconsin loess samples having plasticity indices greater than 20 percent do not need to be treated with hydrogen peroxide, as specified in ASTM and AASHO methods, providing the organic matter content is low. If sodium metaphosphate is used as the deflocculating agent, the hydrochloric acid treatment is not advantageous or desirable for Wisconsin loess samples having high carbonate contents.

To avoid frequent replacement of the standard metal stirring paddle due to wear, the rubber paddle used by the soils laboratory of the Iowa State Highway Commission can be used without loss of stirring efficiency (there may even be a slight increase). A stirring period of four minutes with the high-speed stirring apparatus is adequate for Wisconsin loess samples.

INTRODUCTION

Loess is a fine-grained soil material whose origin, whether aqueous or aeolian, has been a subject of debate among geologists for many years. As a result of this controversy, no single definition of loess has received general acceptance. A comparatively broad definition is:[5]

> Loess, then, is a buff-colored, nonindurated sedimentary deposit consisting predominantly of particles of silt size. Commonly it is nonstratified, homogeneous, calcareous, and porous, and it may possess a weak vertical structure resembling jointing.

Most geologists have been won over to the aeolian concept, which contends that the bulk of the loess was transported and deposited by wind action. Fine sediments exposed without vegetative cover to the winds in desert basins, river flood plains, glacial drift and outwash surfaces are considered to be the major sources of loess.

Surface deposits of loess cover extensive areas in the United States[6] as well as in other parts of the world[7]. Thicknesses of the deposits vary from a few feet to over 1,000 feet, and the loess is not usually intermixed with soil materials of different origin. Approximately two-thirds of the state of Iowa has a surface covering of Wisconsin loess of variable thickness. This loess, also called Peorian loess, blankets older loesses and pre-Wisconsin Pleistocene deposits and is named after the Wisconsin glaciations from which the loess presumably originated. It has been estimated that, if all the Wisconsin loess in Iowa were evenly distributed over the state, it would average about 10 feet in depth. The deepest sections of Wisconsin loess are found in counties bounding the western side of the state, along the east valley wall (first bluff line) of the Missouri River. Here, where the wide flood plain and persistent sand bars of the great river have maintained an ever-replenished supply of dust and silt, the loess has accumulated to thicknesses of from 60 to over 100 feet.

A special study, made in connection with the southwestern Iowa loess investigation, had as its purpose the development of a procedure for dispersing Wisconsin loess for mechanical analysis.

The ASTM (D422-39) and AASHO (T88-49) standard methods of mechanical analysis [1, 11] recommend definite procedures for dispersing soil. These procedures were developed for use with a wide variety of soils; however, it seemed probable that they would not give optimum dispersion for loess soils, especially those containing large amounts (up to 18 percent $CaCO_3$ by weight) of carbonates. (Carbonate contents are expressed as percentages of calcium carbonate, $CaCO_3$, by weight.) Many investigators in the fields of agriculture, ceramics, geology, and engineering have studied the problems of soil dispersion.

PRINCIPLE OF SOIL DISPERSION

An accurate and reproducible mechanical analysis depends on the complete and stable dispersion of a soil sample. In other words, all soil aggregates (secondary particles) must be separated as nearly as possible, into primary particles, and these particles must then be kept in a dispersed state throughout the mechanical analysis. It is necessary to understand, therefore, not only the factors causing aggregation in soils but also those which may cause the flocculation of primary particles once they have been separated.

Causes of Aggregation

Soil aggregates are formed primarily as a result of the presence in soil of the colloidal cementing agents, such as clay, organic matter, and oxides of iron and alumina. The cementation effect of colloidal clay is believed to be the result of dehydration of the clay particles. When the moisture content of a soil is fairly high, the water films surrounding clay particles are so thick that the cohesive forces between adjacent particles are not significant. As the moisture content decreases, the water films become thinner and thinner, and the cohesive forces increase accordingly. In the air-dry condition such water films have great bonding strength; rehydration, however, tends to destroy the bond.

Colloidal organic matter has long been recognized as a cementing agent in soils, but there apparently is no exact knowledge of the mechanism of the cementation effect. In the explanations that have been offered by various investigators, most point to some type of oriented adsorption of organic molecules on the surfaces of clay particles[2]. Such adsorption appears to be stabilized by subsequent dehydration of the adsorbed colloidal organic matter. Since rehydration is extremely slow, this process causes a tenacious cementing influence which is responsible for aggregate formation.

Dehydrated oxides of iron and alumina are responsible for aggregate formation in lateritic soils. The cementation effects of these oxides appear to be principally due to the irreversibility of the colloids upon dehydration.

In addition to the cementing agents in some soils carbonates may also contribute a weak cementation effect. Carbonates may be present in soil as a mixture with clay; they may also occur as secondary concretions or as silt-size powders[14]. It appears that the cementation effect is largely related to the carbonates mixed with clay and will probably become insignificant when the clay is hydrated.

Causes of Flocculation

In the dispersion of a soil sample, the elimination of cementation effects is not sufficient to insure a stable soil suspension; since the separated pri-

mary particles may again coalesce into floccules or aggregates. (In the mechanical analysis of soil, the term *soil suspension* refers to the soil-water mixture, which actually is a combination of a colloidal solution and a suspension of noncolloidal particles.) The electric charge, or zeta potential of clay particles, which is predominantly negative, is considered the determining factor in the stability of a soil suspension. If the zeta potential is sufficiently high, the clay particles will repel each other when they collide during their constant haphazard (Brownian) movement in the suspension. If the zeta potential is low, there will be little repulsion, and the particles will coalesce as a result of a collision and settle out as a floccule.

The amount of the negative electric charge depends largely on the combination of the mineralogical nature of the clay particle or mineral and the kind of adsorbed cations (positive ions). For that reason, with each type of clay mineral the kind of adsorbed cations has an important bearing on the degree of dispersion obtained. The dispersive power of the following monovalent cations decreases according to the series: lithium (Li) > sodium (Na) > potassium (K), and ammonium (NH_4). These cations cause the zeta potential of clay particles to be high and thus may be classified as deflocculating cations. Common flocculating cations, which cause the zeta potential to be low, are hydrogen (H), calcium (Ca), and magnesium (Mg), all found in natural soils.

General Dispersion Procedure

The essential requirements for dispersing a soil may be summarized as: first, the elimination of cementation effects, and second, the prevention of flocculation. The removal of the cementing influences of the irreversible or slowly reversible oxides of iron and alumina is a difficult problem for which no generally acceptable solution has been found. But these oxides are in significant amounts only in lateritic soils; therefore their binding effects are usually assumed as negligible in most soils of the United States. The elimination of the cementation effects of the other soil colloids, organic matter and clay, can be accomplished by oxidation of the organic matter followed by rehydration of the clay particles. Flocculation of dispersed clay particles is prevented by replacing flocculating cations with deflocculating cations.

Organic matter in soils can be removed by oxidation with hydrogen peroxide; boiling soils with 6 percent hydrogen peroxide is a generally accepted method. This treatment is highly effective and produces only carbon dioxide and water as the byproducts of oxidation. The oxidation step in the dispersion procedure can be omitted with engineering soils of low organic content.

The rehydration of clay particles is accomplished by soaking the soil sample in distilled water for a specified period of time, followed by mechani-

cal agitation of the soil-water mixture. (A deflocculating agent is added before agitation.) The more important methods of mechanical agitation are: stirring[1, 3, 11], shaking[10], boiling[16], rubbing, and bubbling air through the soil suspension[17]. High-speed stirring with apparatus patterned after the electric malted-milk mixing machine is perhaps the most extensively used method in engineering laboratories; the Wintermyer apparatus is also used.

The prevention of flocculation in a soil suspension is more complicated than the removal of cementing influences. Because of the variable nature of soils, no specific method has been developed which is entirely satisfactory for all kinds. Techniques which are used attempt to control the kind of cations adsorbed on the clay particles so that the zeta potential of the particles will be high. As has been pointed out, clay particles with a high negative charge will repel each other upon collision, and the soil suspension will be in a stable condition.

To control the type of adsorbed cations two things are necessary. The cations which cause flocculation must be replaced with cations that promote deflocculation, and the replaced cations and other free cations which might later interfere with the dispersion must be rendered ineffective. Both of these may be done by the one-step method of adding a deflocculating agent, or by a two-step method which involves a preliminary treatment of the soil sample before adding the deflocculating agent.

In the one-step method, adsorbed flocculating cations are replaced by cations from the dissociated deflocculating agent; the replaced flocculating cations, together with other free cations in the soil suspension, are made ineffective either by precipitation as insoluble compounds or by the formation of soluble but undissociated compounds. This method is simpler and faster than the two-step method, and it has proved satisfactory for many types of soil.

A more complete dispersion may be obtained for some types of soil with certain deflocculating agents by use of the two-step method. In general, soils benefited by the two-step method are those containing appreciable amounts of carbonates. The predominant basic cations in such soils are the strongly adsorbed divalent calcium and magnesium cations. The first step or preliminary treatment in this method involves replacing adsorbed basic cations with hydrogen cations and removing replaced cations and salts, such as carbonates, from the soil. The second step is to replace the hydrogen cations with deflocculating cations. The preliminary treatment can be accomplished either by electrodialysis or by leaching with dilute hydrochloric acid. The electrodialysis technique is not used as extensively as the acid treatment because it requires additional expensive equipment[8]. In the acid treatment, 0.2N hydrochloric acid is commonly used to leach the soil sample. The sample then must be washed free of electrolytes[9]. The complete removal of chlorides is of special importance.

The steps used in dispersing soil for mechanical analysis will depend on the nature of the soil, the kind of deflocculating agent used, and the fundamental objective of the mechanical analysis (table I).

TABLE I. GENERAL PROCEDURE FOR DISPERSING SOIL FOR MECHANICAL ANALYSIS

Step	Operation	Principal Purpose	Remarks
1	Hydrogen peroxide treatment	To remove organic matter	May be omitted for soils having low organic matter contents
2	Acid treatment (followed by washing)	To replace basic cations with hydrogen and to remove carbonates	Necessary only for some soils with certain deflocculating agents
3	Soaking in water	To rehydrate clay particles	Soaking period of at least 18 hours
4	Addition of deflocculating agent	To prevent flocculation by increasing the zeta potential	Deflocculating agent is added in solution form
5	Mechanical agitation	To effect thorough dispersion of the primary soil particles	High-speed stirring is the conventional method

Deflocculating Agents

Sodium compounds, which dissociate to furnish sodium cations, are the most extensively used deflocculating agents; the higher cost of lithium compounds makes them impractical to use.

A comparison of the efficiency of several deflocculating agents for the dispersion of soil samples from four different states showed that sodium silicate is the most satisfactory deflocculating agent[12]. In the ASTM and AASHO standard methods of mechanical analysis, 20 ml. of 3 degree Baumé sodium silicate solution (prepared by dissolving sodium metasilicate crystals ($Na_2SiO_3 \cdot 9H_2O$) in distilled water) is used for the purpose of deflocculation.

Sodium oxalate ($Na_2C_2O_4$) is a satisfactory deflocculating agent for many types of soil[9], and sodium pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$) is the best for dispersing several types of clay[15].

Sodium metaphosphate ($Na_4P_4O_{12}$) is an excellent deflocculating agent for a wide variety of soils[13]. The effectiveness of sodium metaphosphate was explained as due to a more or less complete replacement of flocculating cations, particularly calcium, by sodium cations from the deflocculating agent. Since the exchange of cations results in the formation of a soluble but highly undissociated sodium calcium metaphosphate complex, the activity of calcium in a soil suspension is eliminated, and soil particles will remain in a dispersed state. If calcium carbonate colloids are present in a soil suspension, the dispersion of these colloids will also be stabilized due

to the influence of sodium metaphosphate. This stabilization is probably due to the coating of a gelatinous substance over the calcium carbonate colloids.

DEVELOPMENT OF DISPERSION PROCEDURE FOR LOESS

The review of the principle of dispersion served as a background and guide for the development of a dispersion procedure especially adapted to the Wisconsin loess of southwestern Iowa. In addition, experiments were performed to evaluate several deflocculating agents, to determine the need for hydrogen-peroxide and hydrochloric-acid treatments, and to evaluate a rubber stirring paddle and to determine stirring-time requirements.

As a part of the southwestern Iowa loess investigation, more than 100 loess samples have been taken in the deep Wisconsin loess area, which includes the whole or parts of twenty counties[4]. This wind-blown soil material is thought to be associated with the glaciations of the Wisconsin stage. Major sources of supply were perhaps the raw surfaces of the newly deposited drifts in northwestern Iowa and the flood plains of the Missouri River and its tributaries which drained the drift areas.

The Wisconsin loess thicknesses in the southwestern Iowa area vary from over 100 ft. along the west boundary (east valley wall of the Missouri River) to about 17 ft. on hilltops near the arbitrary east boundary. In addition to the decrease in thickness with increasing distance from the valley wall, the loess becomes finer textured and its carbonate content decreases from a high of 18 percent ($CaCO_3$ by weight) along the west boundary to a low of 1 percent at the east.

Two loess samples were selected for the dispersion experiments to represent the extreme ranges in physical and chemical property variations; Sample A was taken at the west boundary and Sample B at the east. Both samples were obtained at a depth of 2 to 3 ft. below the top of the C horizon; Sample A was secured in the Hamburg pedological series and Sample

TABLE II. PROPERTIES OF WISCONSIN LOESS SAMPLES FROM SOUTHWESTERN IOWA

Sample Designation	A	B
Location of Sample	Ridge at West Boundary	Hilltop at East Boundary
Liquid Limit, %	30.9	48.3
Plastic Limit, %	25.2	21.7
Plasticity Index, %	5.7	26.6
Organic Matter Content, % by weight	0.30	0.21
Carbonate Content, % $CaCO_3$ by weight	15.0	1.6

B in the Marshall series. Properties which may influence the dispersion of the two samples are given in table II which shows that the organic matter content of both samples was low. Particle-size distribution curves are shown in figure 1. Differential thermal analyses on the samples indicate that both samples contain the same general kinds of clay minerals, apparently of the illite and montmorillonite types. Further mineralogical studies are being made to determine more definitely the mineralogy of the Wisconsin loess.

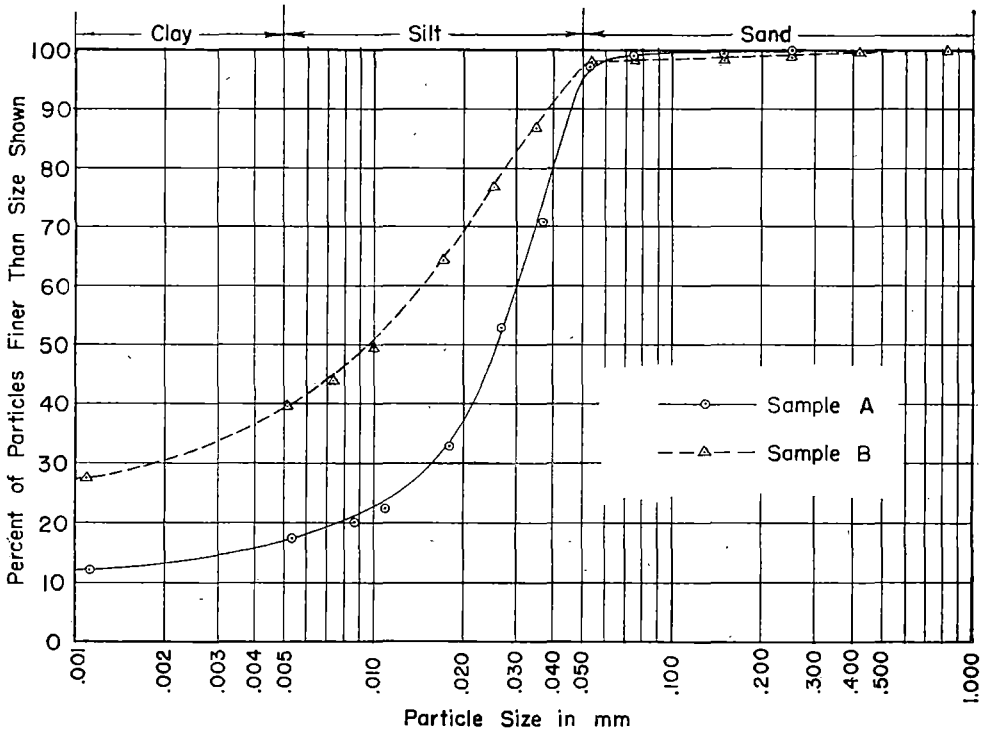

Fig. 1. Particle-size accumulation curves for samples A and B.

Evaluation of Deflocculating Agents

Mechanical analyses were performed for loess samples A and B using solutions of sodium silicate (prepared from sodium metasilicate crystals), sodium oxalate, sodium pyrophosphate and sodium metaphosphate in varying amounts as deflocculating agents. (The sodium metaphosphate solution was prepared from the commercial product *Calgon*, in which the sodium metaphosphate is buffered with a small amount of sodium carbonate to make it stable.) All four of these chemicals have been used by other investigators and have been found promising as deflocculating agents[9, 12, 13, 15]. The

concentrations used in this experiment are shown in table III. The 3 degree Baumé solution of sodium silicate is specified by ASTM and AASHO. A concentration of 0.5N was chosen for the other three chemicals for purposes of direct comparison and convenience of preparation.

TABLE III. EFFECT OF TYPE AND AMOUNT OF DEFLOCCULATING AGENT ON THE EFFICIENCY OF DISPERSION

Deflocculating Agent			Sample A		Sample B	
Type	Conc. of Solution	Amount* (ml.)	Percent Finer Than .005 mm.	Percent Finer Than .001 mm.	Percent Finer Than .005 mm.	Percent Finer Than .001 mm.
Sodium Silicate	3° Baumé	10	12.0	4.8	32.7	17.2
		20	12.2	5.8	34.3	18.0
		30	12.2	6.0	33.0	17.6
		40	12.0	6.0	33.3	18.8
		50	12.6	6.0	32.8	18.0
Sodium Oxalate	0.5 N	10	14.0	7.6	35.0	20.0
		20	14.1	8.0	36.6	24.0
		30	14.2	Flocculated	37.8	26.0
		40	12.2	Flocculated	38.4	27.0
		50	10.2	Flocculated	39.0	27.5
		60			39.4	27.4
		70			39.8	26.4
Sodium Pyrophosphate	0.5 N	10	13.6	8.0	34.6	20.2
		20	16.0	10.0	36.5	23.5
		30	17.1	10.8	38.2	25.2
		40	17.4	10.8	39.2	26.1
		50	16.9	10.5	39.6	26.6
		60	16.0	10.1	39.6	26.9
		70			39.2	27.0
		80			38.3	26.8
Sodium Metaphosphate †	0.5 N	10	14.2	8.6	34.8	20.6
		20	16.4	10.0	36.4	23.0
		30	17.2	10.6	37.3	24.8
		40	17.5	11.3	38.1	26.0
		50	17.4	11.9	38.8	26.8
		60	17.0	12.0	39.2	27.4
		70	18.0	10.6	39.6	27.9
		80			39.8	28.1
		90			39.7	28.0
		100			39.4	27.9
		110			39.2	27.8
		120			39.8	27.8
No Deflocculating Agent			11.7	1.6	32.6	15.2

*Refers to the amount of deflocculating solution used in dispersing a sample of 65 grams into a one liter soil suspension.

†In preparing the 0.5 N solution of sodium metaphosphate, *Calgon* was considered to have the gram-molecular weight of pure sodium metaphosphate.

The only variable in the mechanical analyses was the type and amount of deflocculating solution added to the soil-water mixture prior to high-speed stirring. The hydrometer method was used for determining percentages of 0.005 mm. and 0.001 mm. clay. The efficiency of the deflocculating agents was rated on the basis of the magnitude of these clay size percentages.

The dispersion procedure adopted in this experiment was the following:

1. A 65 gram air-dry loess sample was soaked in distilled water for eighteen hours.
2. The deflocculating agent was added in solution form.
3. The soil-water combination was mixed for 4 min. in a high speed stirring apparatus of the malted milk mixer type.

This procedure is essentially the same as that specified in the ASTM standard method (D422-39), with three modifications: (1) the stirring time was increased from 1 to 4 min; (2) a rubber stirring paddle was substituted for the conventional metal one; and (3) the hydrogen peroxide treatment was omitted for Sample B, which had a plasticity index greater than 20.

The hydrometer test was performed essentially according to the standard ASTM method, except that a correction was applied to the hydrometer

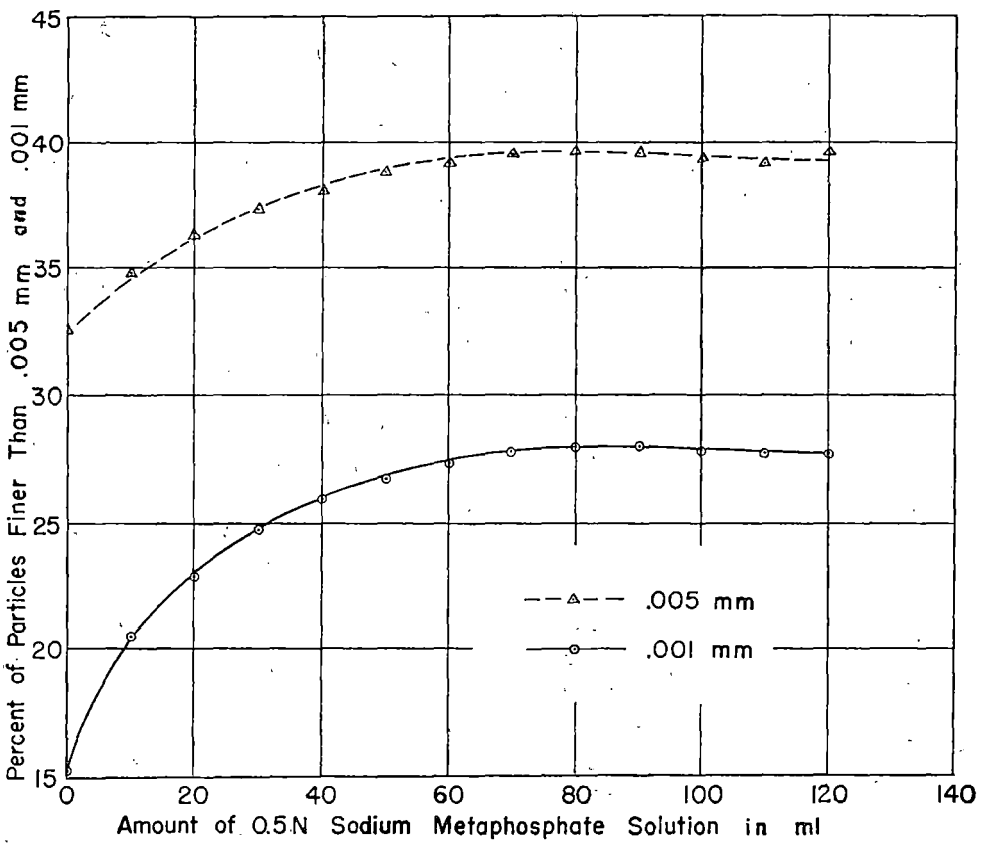

Fig. 2. Relation between amount of sodium metaphosphate and degree of dispersion for sample B.

reading to compensate for the direct influence of the deflocculating agent on the reading.

The effect of type and amount of deflocculating agent on the efficiency of dispersion of samples A and B is shown in table III. The table also shows the amounts of 0.005 mm. and 0.001 mm. clay measured without use of a deflocculating agent (other test steps were the same). The 0.005 mm. and 0.001 mm. clay-size data show a similar trend; this is illustrated graphically by figure 2, which shows the relation between amount of sodium metaphosphate and degree of dispersion for loess sample B. For this reason, the deflocculating agents are compared graphically on the basis of only the 0.001 mm. clay measurements (figures 3, 4).

In a comparison of the efficiency of the four chemicals as deflocculating agents for sample A, sodium metaphosphate and sodium pyrophosphate were superier to sodium oxalate and sodium silicate; the latter chemical

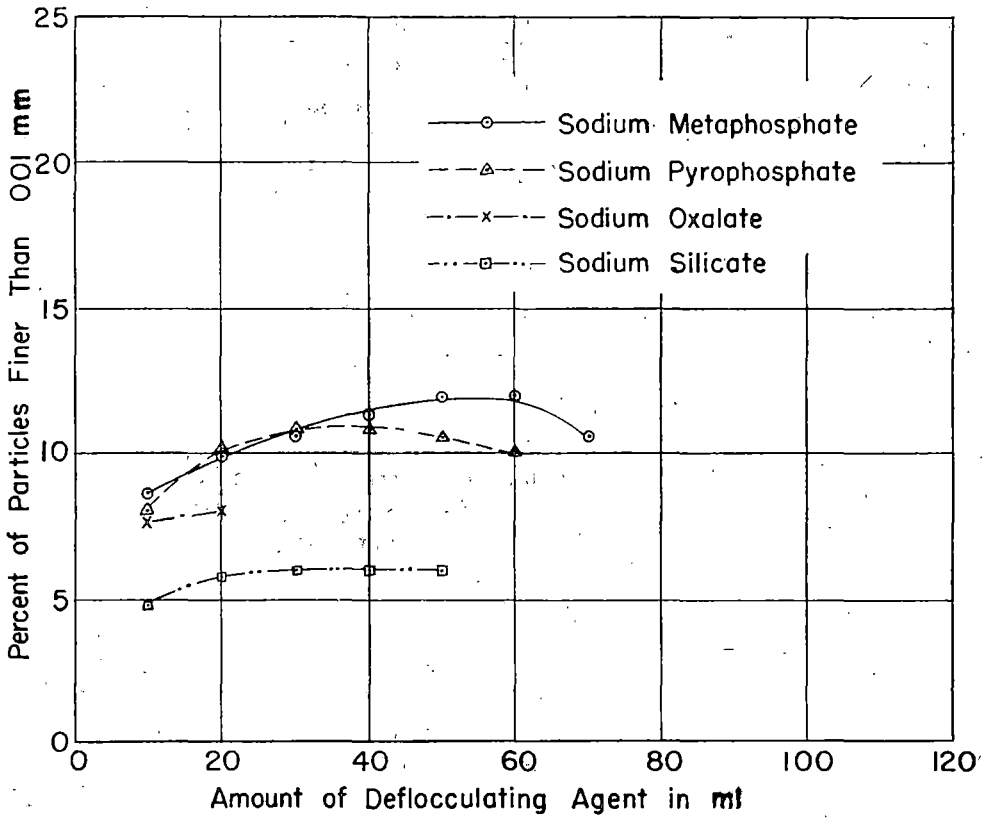

Fig. 3. Comparison of efficiency of deflocculating agents for the dispersion of sample A.

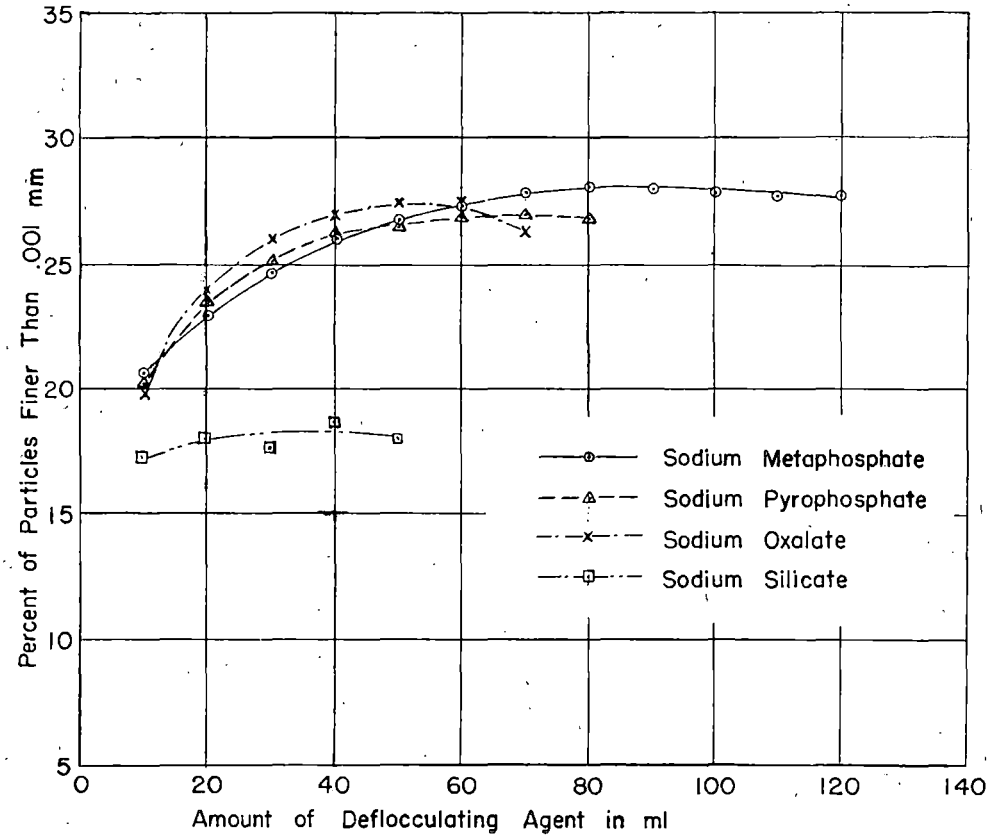

Fig. 4. Comparison of efficiency of deflocculating agents for the dispersion of sample B.

showed the least promise. Flocculation occurred, with additives of sodium oxalate solution greater than 20 ml. sodium metaphosphate rated highest as a deflocculating agent for sample A, with optimum dispersion being obtained with about 60 ml. of 0.5 N solution.

The sodium silicate in sample B was decidedly inferior to the other three chemicals (figure 4). Sodium metaphosphate rated slightly higher than sodium pyrophosphate and sodium oxalate, with 80 ml. of 0.5 N sodium metaphosphate solution giving optimum dispersion. However, if 60 ml. of solution is used instead of 80 ml., only a slight loss in dispersive power is experienced, the decrease in the percent of 0.001 mm. clay measured being only 0.7 percent (figure 4).

In consideration of the moderate degree of reproducibility of the hydrometer analysis, and to develop a uniform dispersion procedure, the 60 ml. of 0.5 N sodium metaphosphate solution was adopted as the deflocculating additive for mechanical analyses of the Wisconsin loess (figure 1).

Additives of sodium silicate solution in excess of 20 ml. do not significantly improve the degree of dispersion of the two loess samples (figures 3, 4). Twenty milliliters is the amount of sodium silicate solution recommended in the ASTM and AASHO dispersion procedures.

Hydrogen Peroxide Treatment

The main purpose of the hydrogen peroxide treatment is, as mentioned previously, to remove the cementing influences of organic matter from soil. Since the organic content is low in all Wisconsin loess samples taken from southwestern Iowa, this treatment appeared to be unnecessary. However, it was considered desirable to determine experimentally the effect of the hydrogen peroxide treatment on sample B (P.I. = 26.6 percent), because the ASTM dispersion procedure specifies that soils having a plasticity index greater than 20 percent shall be treated with hydrogen peroxide (6 percent H_2O_2) for the purpose of aiding dispersion, rather than for the removal of organic matter. The deflocculating agents used for this experiment were sodium metaphosphate (60 ml. of 0.5 N solution) and sodium silicate (20 ml. of 3 degree Baumé solution). Sodium silicate was used because it is the deflocculating agent specified by the ASTM procedure.

The hydrogen peroxide treatment of sample B was performed in accordance with ASTM directions. Hydrometer analyses to determine the percent of particles finer than 0.005 mm. and 0.001 mm. were made as previously in connection with the evaluation of deflocculating agents. The test data indicate that the hydrogen peroxide treatment did not improve the degree of dispersion of sample B, as reflected in the magnitude of the 0.005 mm. and 0.001 mm. clay determinations (table IV). For that reason this treatment is omitted in the dispersion procedure for Wisconsin loess samples which have high plasticity indices but low organic matter contents.

TABLE IV. EFFECT OF HYDROGEN PEROXIDE TREATMENT ON DEGREE OF DISPERSION OF LOESS

	SAMPLE B		
Deflocculating Agent	Hydrogen Peroxide Treatment	Percent Finer Than .005 mm.	.001 mm.
60 ml. 0.5 N Sodium Metaphosphate Solution	Yes	38.6	27.2
	No	39.2	27.4
20 ml. 3° Baume Sodium Silicate Solution	Yes	33.0	16.8
	No	34.3	18.0

Acid Treatment

The value of the hydrochloric acid treatment as an aid to more efficient dispersion was investigated by experiments with highly calcareous ($CaCO_3$ =15.0 percent) loess sample A. The purpose of the acid treatment has previously been discussed and is summarized in table I.

One hundred grams of air-dry loess was soaked in 1,650 ml. of 0.2 N hydrochloric acid, with occasional stirring and shaking, for 24 hours. (Milliliters of 0.2 N HCl = (1.1) (% $CaCO_3$ in soil) (grams of soil). This equation provides an excess of HCl over that needed to remove the carbonates.) At the end of the soaking period, the acid and soil mixture was poured onto filter paper in a Buchner funnel, and the acid was drawn through by means of an aspirator. The soil was then washed with distilled water until the chlorides had been completely removed, as indicated by testing the leachate with silver nitrate; chlorides are considered eliminated if the leachate shows no cloudiness when a few drops of silver nitrate are added. After the removal of the chlorides, the washed sample was air dried prior to preparing 65 grams for the mechanical analysis.

The validity of using the acid treatment in engineering mechanical analyses of calcareous soils is questionable, since the carbonates, an inorganic constituent of the soil, are removed and are not represented in the particle size distribution determination. With a highly calcareous soil, such an omission would misrepresent the soil's texture. This fact, and the added time required for the acid treatment, makes it advantageous to use a deflocculating agent whose dispersive power is not impaired by the presence of carbonates. The data indicate no significant evidence of increased dispersion with the acid treatment when sodium metaphosphate is used as the deflocculating agent (table V). It was, therefore, considered inadvisable to include the acid treatment in the dispersion procedure for Wisconsin loess.

TABLE V. EFFECT OF ACID TREATMENT ON DEGREE OF DISPERSION OF LOESS

	SAMPLE A*	
Acid Treatment	Percent of Particles Finer Than .005 mm.	.001 mm.
Yes	17.2	13.0
No	17.0	12.0

*60 ml. of 0.5 N sodium metaphosphate solution was used as the deflocculating agent.

Type of Stirring Paddle

The high speed electric stirring apparatus of the ASTM and AASHO standard methods of mechanical analysis utilizes a replaceable metal paddle which is subject to rapid wear by the abrasive action of hard soil grains in the soil water mixture. Frequent replacement of the paddle is necessary

to insure a standard intensity of stirring. Because of the wear factor, the soils laboratory of the Iowa State Highway Commission has for several years been using a wear resistant rubber paddle with highly satisfactory results. The square rubber paddle, cut from an automobile inner tube, is 7/8 in. on each side and 1/8 in. thick and is mounted on the stirring shaft in the same manner as the metal paddle.

A series of mechanical analyses were performed on samples A and B to compare the stirring efficiencies of the rubber and metal paddles. Sodium metaphosphate (60 ml. of 0.5 N solution) was the only deflocculating agent used. The data indicate that the rubber paddle compares favorably with the metal paddle and possibly may give even slightly better disperson (table VI). As a result of this experiment, the rubber paddle was adopted for use with the Wisconsin loess.

TABLE VI. EFFECT OF TYPE OF STIRRING PADDLE AND LENGTH OF STIRRING PERIOD ON DEGREE OF DISPERSION*

Type of Stirring Paddle	Stirring Period (min.)	Sample A Percent Finer Than .005 mm.	Sample A Percent Finer Than .001 mm.	Sample B Percent Finer Than .005 mm.	Sample B Percent Finer Than .001 mm.
Standard Metal Paddle	1	14.0	9.3	36.3	24.5
	4	15.5	10.6	37.4	25.7
Square Rubber Paddle	1	14.8	9.6	38.0	25.5
	4	17.0	12.0	39.2	27.4
	10	17.2	12.0	39.5	27.9
	16	17.4	12.8	39.6	28.7
No Stirring	0	0	0	7.0	0

*60 ml. of 0.5 N sodium metaphosphate solution was used as the deflocculating agent.

Length of Stirring Period

The length of the stirring period used with the high speed malted milk mixer type apparatus is an important part of the dispersion procedure. The optimum stirring time should give maximum dispersion of the soil water mixture on the one hand, and minimum degradation (break-up of the primary soil particles into smaller ones) on the primary soil particles on the other. A long stirring period is desirable for a high degree of dispersion, but it is objectionable if degradation occurs.

The amount of degradation during a given stirring period depends largely upon both the soil texture and the hardness of the individual particles. The coarse sand fraction (2.0 mm. to 0.42 mm.) is most affected by degradation during the stirring operation[17]. The coarse sand fraction in Wisconsin loess (figure 1) is negligible; and since quartz is believed to be the predominant mineral in the nonclay sizes, the soil particles most subject to degradation are likely to be extremely hard. Because of this, the

action of degradation is probably of minor importance in the loess.

The effect of length of stirring period on the degree of dispersion is shown in table VI and in figure 5. For both samples A and B the increase in the percentage of particles finer than 0.005 mm. and 0.001 mm. is very slight for increases in stirring time above four minutes.

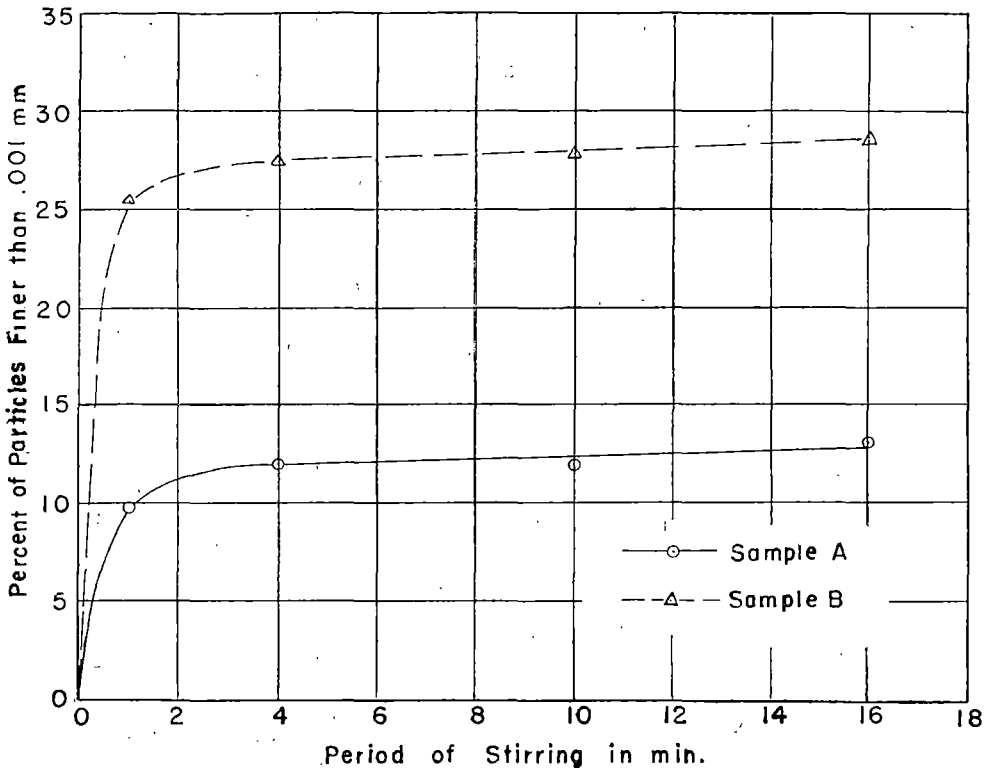

Fig. 5. Relation between length of stirring period and degree of dispersion.

A one minute stirring period is specified in both the ASTM and AASHO dispersion procedures, presumably to avoid the degradation which may result from a longer stirring time. Since the action of degradation appeared to be of minor importance in stirring loess, especially with a rubber paddle, a stirring period of four minutes was adopted for use with the Wisconsin loess. The soils laboratory of the Iowa State Highway Commission uses the four minute stirring period with all types of Iowa soils.

CONCLUSIONS

1. The use of deflocculating agents other than sodium silicate, which is specified in the ASTM and AASHO standard methods of mechanical analysis, results in a more complete dispersion of the Wisconsin loess of southwestern Iowa.

2. Among the deflocculating agents which were compared experimentally, sodium metaphosphate is the most efficient in dispersing the Wisconsin loess. The amount of sodium metaphosphate adopted for dispersing Wisconsin loess samples for mechanical analysis determinations is 60 ml. of 0.5 N solution.

3. Wisconsin loess samples having plasticity indices greater than 20 percent do not need to be treated with hydrogen peroxide, providing the organic matter content is low.

4. If sodium metaphosphate is used as the deflocculating agent, the hydrochloric acid treatment is not advantageous or desirable for Wisconsin loess samples having high carbonate contents.

5. To avoid frequent replacement of the standard metal stirring paddle due to wear, the rubber paddle used by the soils laboratory of the Iowa State Highway Commission can be used without loss of stirring efficiency; there may even be a slight increase in stirring efficiency.

6. A stirring period of four minutes with the high speed malted milk mixer type stirring apparatus is adequate for Wisconsin Loess samples.

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SIMPLIFIED AIR-JET DISPERSION APPARATUS FOR MECHANICAL ANALYSIS OF SOILS

by

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(Highway Research Board Proceedings, 32:541-547. 1953.)

ABSTRACT

The simplified air-jet dispersion apparatus is compared with three other dispersion apparatus in current use. Results of mechanical analyses on a wide variety of soils indicate that the new device gives a comparatively high degree of dispersion without causing significant degradation. This apparatus is simple in construction and can be built at a relatively low cost. Because of its unique design the procedure for its use is substantially simpler than that for other dispersion apparatus.

Mechanical analysis to determine particle-size distribution in soils is a common test used in civil engineering, ceramic engineering, agricultural, and geological testing laboratories. An essential step in the mechanical analysis procedure is to disperse the soil sample in water so there are no aggregated or flocculated particles to distort test results. This is usually accomplished by soaking the sample in water for a prescribed length of time and then subjecting it to mechanical agitation in the presence of a deflocculating agent.

REVIEW OF SOIL DISPERSION METHODS

Different types of apparatus have been used to disperse soil for mechanical analysis, but none of them has been found entirely satisfactory. Because of this, a project (Project 300) was established at the soil research laboratory of the Iowa Engineering Experiment Station to investigate the possibility of developing a new dispersion apparatus which would be simple in construction, easy to use, and would give the desired dispersion.

Various techniques have been used to agitate a soaked soil sample for achieving proper dispersion. Among the ones more commonly used are end-over-end shaking, stirring with a high speed, electric malted milk mixer, and vigorous agitation by jets of compressed air.

End-over-end shaking of a soaked soil sample in a glass tube or jar is one of the oldest methods for soil dispersion[5]. It is still widely used in the fields of agriculture, geology, and ceramic engineering. The machine used for shaking rotates at a slow speed, usually 40 to 70 revolutions per minute. This method gives fairly satisfactory results with many types of soil; its main disadvantage is that the period of dispersion is long, usually 24 hours or more.

Both the American Society for Testing Materials and the American Association of State Highway Officials have adopted the electric malted milk mixer for stirring a soaked sample as the apparatus for soil dispersion with a stirring time of one minute[1, 2, 4]. High speed stirring by the use of such apparatus is fairly effective with common types of soil, but with other soils it fails to achieve proper dispersion. Though the effectiveness of dispersion may be improved by allowing a longer stirring period, such prolonged stirring is not advisable because of accompanied increase in the degradation (the breaking up or wearing down of primary soil particles into smaller ones during dispersion) of soil particles[7]. The stirring paddle is usually made of metal, but rubber paddles have also been used[3].

Compressed air directed through either jets or holes agitates a soaked sample in the Wintermyer soil dispersion cup[7]. It gives satisfactory dispersion with a wide variety of soils without causing significant degrading. However, the apparatus is intricate and costly to build, and the procedure for soil dispersion requires a comparatively long time. This apparatus has been adopted by AASHO as an alternate for soil dispersion[6].

DEVELOPMENT OF SOIL DISPERSION TUBE

The Wintermyer soil dispersion apparatus appeared to be satisfactory for use with a wide variety of soils. Because of this, the air jet principle was used in the development of a simplified apparatus, called the soil dispersion tube. The soil dispersion tube has two components, the tube and a glass hydrometer jar (figure 1).

In developing the soil dispersion tube, various designs were tried out in the laboratory. The one shown was found most satisfactory. An important feature of this design is that the tube fits into standard AASHO and ASTM hydrometer jars. This enables the whole hydrometer test, including soaking and agitation, to be carried out in the same jar. In other words, repeated transfer of the soil water mixture from one container to another as required in other dispersion methods is not necessary.

Before developing a procedure for using the tube, the amount of soil water mixture to be used during dispersion, the duration of dispersion, and the amount of air pressure needed to achieve optimum results were determined. Mechanical analyses were performed to compare the effects caused by variations in the amount of soil water mixture, in the dispersion period,

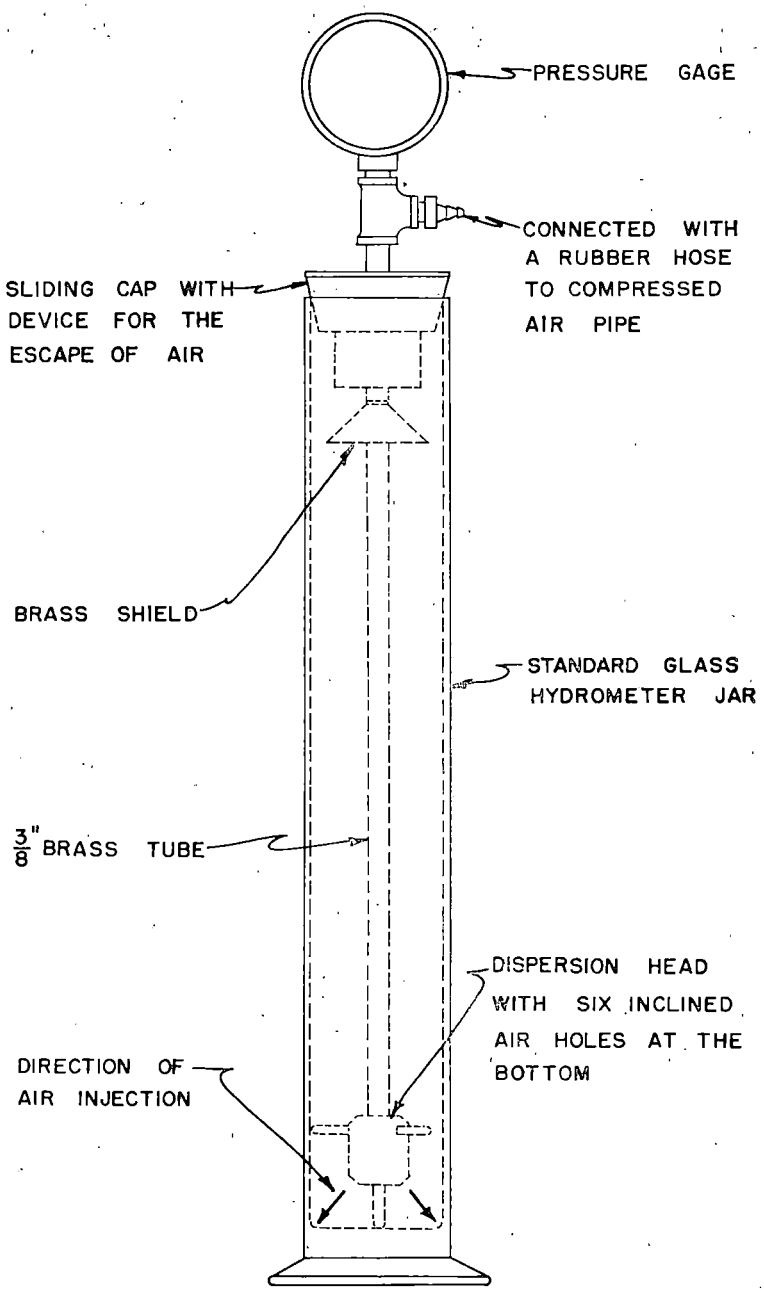

Fig. 1. Soil dispersion tube for mechanical agitation of soil water mixtures.

and in the air pressure. Sandy, silty, and clayey soils collected from different parts of the United States were used in these tests.

Results indicate that the optimum amount of soil water mixture for dispersion is 250 ml. Conclusions regarding dispersion periods and air pressures are that a pressure of 25 psi and a dispersion period of 5 minutes should be used to disperse silty and clayey soils and a pressure of 10 psi and a dispersion period of five minutes should be used to disperse sandy soils.

A tentative procedure for using the soil dispersion tube in the hydrometer method for particle size determination is suggested in the appendix. The tube can also be adapted to other methods of particle size measurement[4].

EVALUATION OF SOIL DISPERSION TUBE

Different types of apparatus may give different degrees of dispersion and may cause varying amounts of degradation. In dispersing a soil sample for mechanical analysis, a high degree of dispersion and a minimum amount of degradation are desired; therefore, the degree of dispersion and the amount of degradation obtained with different dispersion apparatus may be used as criteria for comparing these apparatus.

When a soil sample containing sand, silt, and clay size material is dispersed for particle size determinations, the results obtained will reflect both the degree of dispersion and the extent of degradation. For the comparison of the degree of dispersion and of the extent of degradation, soil samples which are more sensitive to one than to the other should be used.

Since clayey soils are probably more sensitive to the dispersion factor than to the degradation factor, they can best be used to study the degree of dispersion. The degree of dispersion obtained with different apparatus may then be rated on the basis of particle size measurements, particularly the 0.005 mm. and 0.001 mm. sizes. For example, the higher the content of material finer than 0.005 mm. or 0.001 mm., the higher the degree of dispersion.

Sandy soils which have been washed free of silt and clay size material may be used for determining the effects of degradation. The use of a washed sand sample eliminates, for the most part, the dispersion factor. With such a sample, the degradation of sand size material caused by the use of different apparatus may be compared on the basis of sieve analysis results. In general, the larger the amount of material passing each sieve, the greater the degradation. The comparison of degradation on the basis of sand size material is purely a matter of convenience, since the degradation of silt size and clay size materials is comparatively difficult to evaluate.

In evaluating the soil dispersion tube, mechanical analyses were performed on soil samples dispersed by the tube as well as the three types of dispersion apparatus in current use. The procedure for the use of the malted

milk mixer type apparatus, referred to later as the ASTM stirring apparatus, is given in both the ASTM and AASHO standard methods[1, 6]. The end-over-end shaker used in the evaluation study rotates at a rate of 65 revolutions per minute. With this apparatus, soaked soil samples were shaken for 24 hours. The Wintermyer soil dispersion cup B was used in all comparisons[6].

TABLE I. SOURCE AND PROPERTIES OF SOIL SAMPLES*

Sample No.	Source	Textural† Classification	Plasticity Index	Remarks
1	Iowa	Clay	51.7	
2	Virginia	Clay	35.3	
3	California	Clay	38.7	
4	New York	Clay	13.1	
5	Texas	Clay Loam	3.6	
6	Iowa	Silty Loam	6.2	
7	New York	Sandy Loam	NP	
8	Virginia	Sand	NP	High content of mica
9	Iowa	Sand	NP	
10	Iowa	Sand	NP	Silt and clay removed by washing
11	Iowa	Sand	NP	Silt and clay removed by washing

*Only material passing No. 10 sieve was used in this study.
†Textural classifications are based upon the Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction.

A large number of soil samples obtained from different parts of the United States were used for comparing the various types of dispersion apparatus. Reported are results obtained with 11 samples representing soils which are difficult to disperse or highly susceptible to degradation (table I). Four clayey soils, samples 1 to 4, were selected to rate apparatus on the basis of degree of dispersion. Two washed sands, samples 10 and 11, were used for comparing the amount of degradation caused by the different apparatus. The other five soil samples were used for general comparison of the effectiveness of dispersion.

In dispersing all soil samples for mechanical analyses, 20 ml. of 3 degree Baumé sodium silicate solution as specified in AASHO and ASTM standard methods was used as the deflocculating agent.

Table II summarizes the results of mechanical analyses on the 11 samples. Particle size measurements on samples 1 to 4, especially the 0.001 mm. values, indicate that the soil dispersion tube and the Wintermyer soil dispersion cup give the highest degree of dispersion; the tube rates slightly higher. The difference in the degree of dispersion in sample 3 is illustrated graphically (figure 2). Note that the greatest differences in particle size measurements are in the amount of material finer than 0.001 mm.

Results of sieve analyses on samples 10 and 11 (table II) indicate that the tube and the Wintermyer cup cause the least amount of degradation. Sample 11 is a sand which is extremely susceptible to degradation. Particle

TABLE II. MECHANICAL ANALYSIS OF ELEVEN SAMPLES DISPERSED BY DIFFERENT APPARATUS

Sample No.	Weight of test Sample (gm.)	Type of Dispersion Apparatus*	Percent of Particles Finer Than†							
			2.0 mm. (No. 10 Sieve)	0.84 mm. (No. 20 Sieve)	0.42 mm. (No. 40 Sieve)	0.25 mm. (No. 60 Sieve)	0.149 mm. (No. 100 Sieve)	0.074 mm. (No. 200 Sieve)	0.005 mm.	0.001 mm.
1	50	A					100.0	97.4	57.0	34.8
		B					100.0	98.6	57.7	33.4
		C					100.0	98.8	63.0	48.5
		D					100.0	98.8	63.1	51.1
2	50	A			100.0	95.9	91.6	84.1	51.8	25.4
		B			100.0	96.2	91.7	84.8	54.5	40.0
		C			100.0	96.1	91.7	84.7	56.4	43.9
		D			100.0	96.7	92.7	86.2	58.6	45.9
3	50	A	100.0	98.3	95.9	94.1	92.3	89.5	43.9	16.8
		B	100.0	98.4	96.2	94.4	92.6	89.8	52.4	29.3
		C	100.0	98.3	96.1	94.3	92.5	89.7	52.9	38.9
		D	100.0	98.2	95.9	94.2	92.5	89.8	53.7	40.8
4	50	A	100.0	99.0	98.0	97.1	96.2	94.0	55.3	27.3
		B	100.0	99.2	98.2	97.3	96.4	94.1	52.5	26.1
		C	100.0	99.0	98.0	97.1	96.2	94.0	55.1	28.5
		D	100.0	99.3	98.5	97.7	96.9	94.9	56.9	29.8
5	50	A	100.0	99.2	97.6	92.7	82.5	65.8	20.1	2.6
		B	100.0	98.9	97.1	92.3	82.5	66.5	24.0	4.5
		C	100.0	98.8	97.1	92.1	82.1	65.7	21.8	4.6
		D	100.0	99.3	98.1	93.9	85.1	70.4	28.8	7.1
6	50	A					100.0	99.3	11.3	4.2
		B					100.0	99.3	13.6	7.3
		C					100.0	99.4	12.9	6.2
		D					100.0	99.4	17.8	10.4
7	100	A	100.0	97.3	90.5	78.3	59.3	38.6	10.0	2.1
		B	100.0	95.5	89.3	78.7	61.2	40.6	11.3	5.0
		C	100.0	97.1	90.3	78.2	59.2	38.5	11.2	4.0
		D	100.0	95.1	88.6	78.1	60.8	40.6	10.6	3.8
8	100	A	100.0	99.1	71.4	50.2	42.0	25.6	3.8	1.8
		B	100.0	97.6	66.9	44.2	37.1	24.5	5.6	4.0
		C	100.0	96.9	64.3	41.1	33.7	21.0	3.8	1.3
		D	100.0	97.6	64.4	41.0	34.1	21.5	4.8	2.7
9	100	A	100.0	98.9	79.6	38.0	19.9	16.0	3.0	1.2
		B	100.0	98.8	81.8	41.4	19.5	15.6	3.8	1.9
		C	100.0	98.8	79.1	36.9	18.8	15.0	2.8	1.3
		D	100.0	98.9	79.6	38.2	19.3	15.3	3.5	1.3
10	100	A	100.0	74.6	33.9	15.0	7.6	1.8	Trace	Trace
		B	100.0	74.3	34.6	16.1	8.2	2.8	Trace	Trace
		C	100.0	74.4	34.0	14.8	7.3	1.0	Trace	Trace
		D	100.0	73.4	32.6	14.2	6.8	1.0	Trace	Trace
11	100	A	100.0	72.7	51.9	37.9	27.3	17.0	Trace	Trace
		B	100.0	66.4	42.6	29.8	20.7	10.4	Trace	Trace
		C	100.0	65.0	40.8	27.5	17.6	5.9	Trace	Trace
		D	100.0	65.1	40.8	27.3	17.5	5.7	Trace	Trace

*Type A, ASTM stirring apparatus; Type B, end-over-end shaker; Type C, Wintermyer soil dispersion cup; Type D, soil dispersion tube.
†All percentages are the average of results from duplicate tests.

size accumulation curves for the four dispersion apparatus are plotted only for material retained on the No. 200 sieve (figure 3). The "undispersed" curve represents the gradation of sample 11 unaffected by the degrading action of the dispersion apparatus. By comparing the other curves with it, the approximate amount of degradation caused by each type of apparatus can be obtained. The amount of degradation caused by the soil dispersion tube is small. This would be true especially with common types of soil which are usually much less susceptible to degradation than the sample used.

Among the other five soil samples used for the purpose of general comparison, sample 8 is of special significance. Because of a high mica content, it is quite susceptible to degradation. In the procedure for using the Wintermyer apparatus[6], an exceptionally short dispersion period is specified for samples containing large percentages of mica. Data shown in table II indi-

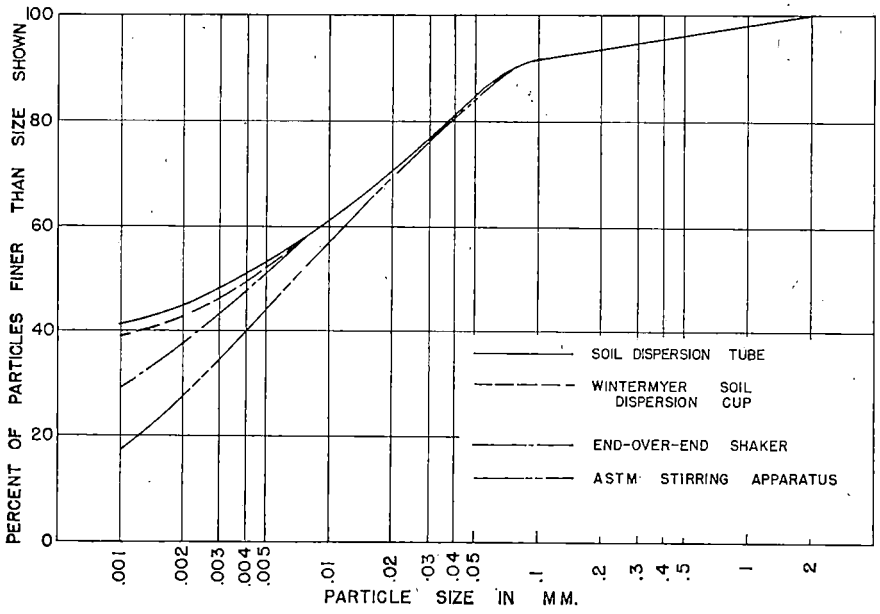

Fig. 2. Particle size accumulation curves for different apparatus, sample 3.

cate that the use of the tube and the Wintermyer apparatus results in approximately the same particle size measurements in the sand fraction of sample 8. Because of this, it is believed that the soil dispersion tube with the regular dispersion period of five minutes may give satisfactory results even for soils with high mica content.

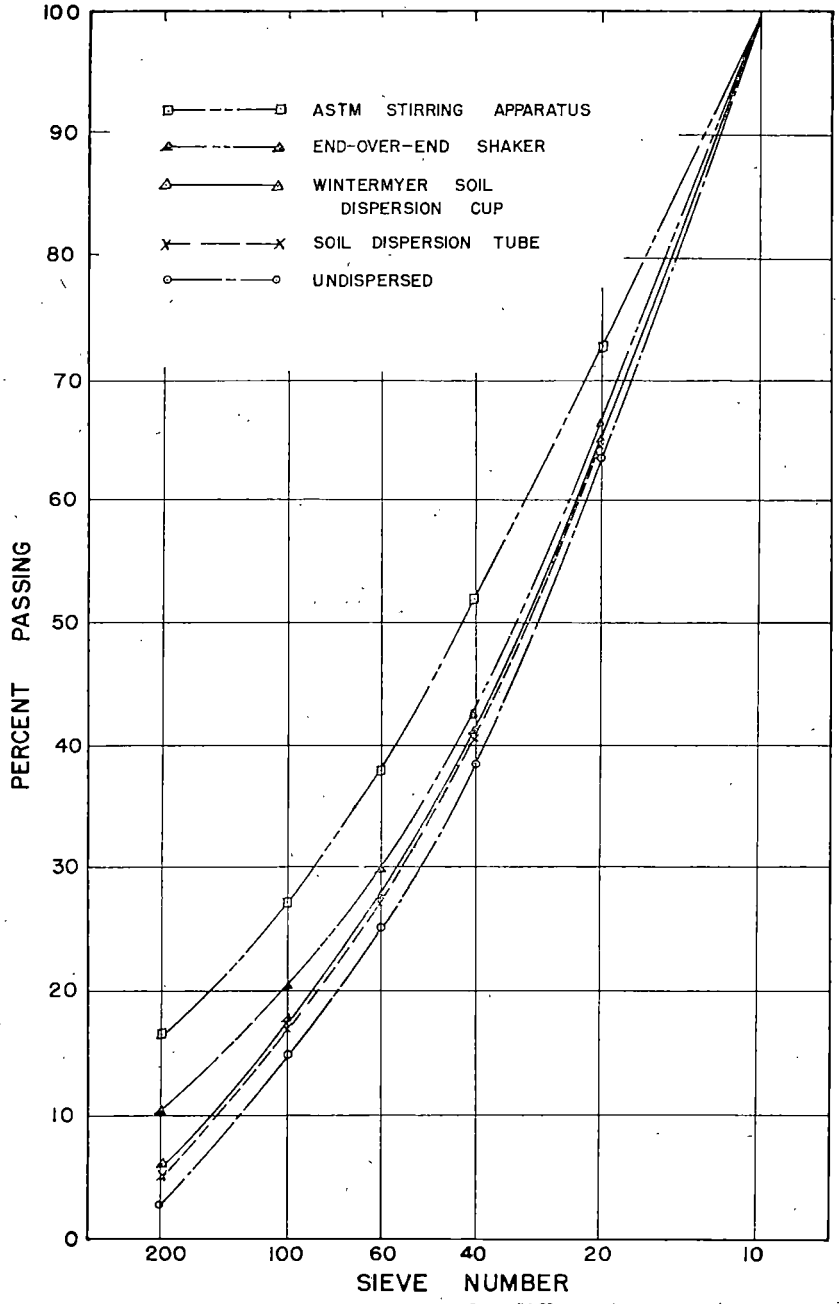

Fig. 3. Particle size accumulation curves for different apparatus, sample 11.

CONCLUSION

1. The soil dispersion tube is a promising dispersion apparatus for mechanical analysis of soils.
2. The comparative experiments presented indicate that the two dispersion apparatus utilizing compressed air, the tube, and the Wintermyer soil dispersion cup, give a comparatively high degree of dispersion without causing significant degradation.
3. Since the tube is a simplified air jet dispersion apparatus, it can be built at a relatively low cost. Because of the unique design of the soil dispersion tube, the procedure for its use is much simpler than that for any other dispersion apparatus in current use.

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APPENDIX

Tentative Procedure For Using The Soil Dispersion Tube To Disperse Soil Samples For Hydrometer Tests

For most soils, a representative sample of 50 gm. shall be secured and placed in a hydrometer jar. Add about 150 ml. of distilled water and stir the soil water mixture thoroughly. After the soil has soaked at least eighteen hours, add a deflocculating agent and a sufficient amount of distilled water to make the resulting mixture approximately 250 ml.

(An alternate soaking procedure is to add the deflocculating agent and a sufficient amount of distilled water to the soil sample to make a mixture of 250 ml. before soaking. Agitate the mixture with the S.D.T. at the end of the prescribed period of soaking.

Both soaking procedures appear to result in the same degree of dispersion. The alternate procedure is convenient to use when different amounts of a deflocculating agent are being tried out.)

Before using the tube, open the control valve on the compressed air pipe until a pressure of about 1 psi is registered on the pressure gauge. (The initial air pressure of 1 psi is required to prevent the soil water mixture from entering the dispersion head of the tube.) Then, insert the tube into hydrometer jar and increase the pressure to 25 psi. At this pressure, the soil water mixture shall be agitated for five minutes.

At the end of the five minute dispersion period, reduce the pressure to 1 psi, lift the tube out of the soil water mixture, and wash all particles clinging to it back into the hydrometer jar. Add more distilled water to the dispersed sample until the mixture attains a volume of 1,000 ml. It is then ready for hydrometer measurements.

For very sandy soils, use a sample weighing 100 gm. and a dispersion pressure of 10 psi; otherwise the procedure is the same.

STUDIES OF DEFLOCCULATING AGENTS FOR MECHANICAL ANALYSIS OF SOILS

by

T. Y. Chu, Assistant Professor, Civil Engineering

D. T. Davidson, Professor, Civil Engineering

(Highway Research Board Bulletin 95. 1954.)

DEFLOCCULATING AGENTS

Adequate and stable dispersion of a soil sample is important for an accurate mechanical or particle size analysis. Since most soils are difficult to disperse in water and tend to flocculate after being dispersed, the chemicals used as deflocculating or dispersing agents are added to the soil water mixture to obtain satisfactory dispersion.

The addition of a deflocculating agent to a soil water mixture affects the degree of dispersion of the soil sample and may also affect the specific gravity of the soil particles and the viscosity and specific gravity of the suspending medium. The experiments discussed in this paper were conducted for the following purposes:

To compare the effectiveness of several chemicals as deflocculating agents for the dispersion of soils;

To determine the effect of one of the deflocculating agents on the specific gravity of the soil dispersed and on the viscosity and specific gravity of the suspending medium.

Soil samples from different parts of the United States were used in the experiments (table I).

Effectiveness

The theory of soil dispersion has been discussed[3]. The effectiveness of a deflocculating agent can be rated on the degree of dispersion of a soil sample with the deflocculating agent. The degree of dispersion can be determined especially by particle size measurements of the fractions finer than 0.005 mm. and 0.001 mm. For example, the higher the content of material finer than 0.005 mm. and 0.001 mm., the higher the degree of dispersion.

Two types of dispersion apparatus were used in the mechanical analysis experiments reported herein: The first is the mechanical stirrer specified by both the American Society for Testing Materials and the American

TABLE I. SOURCE AND SOME PROPERTIES OF SOIL SAMPLES*

Sample No.	Source	Textural Classification†	Plasticity Index	Organic Matter Content, Percent	Content of Carbonates, Percent	pH
1	Iowa	Clay	51.7	1.2	2.5	5.3
2	Virginia	Clay	35.3	0.7	2.5	6.7
3	California	Clay	38.7	0.3	6.8	8.5
4	New York	Clay	13.1	0.6	14.9	8.1
5	Texas	Clay loam	3.6	0.2	81.3	8.2
6	Iowa	Silty loam	6.2	0.3	11.6	8.3
7	Virginia	Sand	N.P.	0.3	40.8	7.4
8	Texas	Clay	42.4	0.3	13.6	7.5

*Only material passing No. 10 sieve was used in this study.
†Textural classifications are based upon the Bureau of Public Roads system except that 0.074 mm. was used as the lower limit of the sand fraction.

Association of State Highway Officials; the second is the Soil Dispersion Tube (S.D.T.). The dispersion procedure with the mechanical stirrer is given in the ASTM and AASHO standard methods of mechanical analysis[4, 5]. The S.D.T. apparatus and its use have been described[1].

Hydrometer tests were performed according to the standard methods of mechanical analysis except that, to compensate for the change in the specific gravity of the suspending medium due to the addition of a deflocculating agent, corrections were applied to hydrometer readings. The determination of correction constants is discussed later in this paper. Particle size measurements reported in this paper are the average of results from duplicate tests.

COMPARISON OF SODIUM SILICATE, SODIUM PYROPHOSPHATE, AND SODIUM METAPHOSPHATE AS DEFLOCCULATING AGENTS

Sodium silicate is specified as the deflocculating agent in the ASTM and AASHO standard methods of mechanical analysis. Sodium pyrophosphate and sodium metaphosphate have been found effective as deflocculating agents for many types of soil[3, 7, 8]. These three chemicals were evaluated as deflocculating agents for the soil samples listed in table I.

In all experiments reported, deflocculating agents in solution were added to the soil water mixture. The concentration of sodium silicate solution prepared from sodium metasilicate crystals was 3° Baumé. The solution of sodium pyrophosphate, also known as tetrasodium pyrophosphate, $Na_4 P_2 O_7 10H_2O$, and of sodium metaphosphate was 0.5 N (table II).

TABLE II. COMPARISON OF EFFECTIVENESS OF DEFLOCCULATING AGENTS IN SOIL DISPERSON

Dispersion Apparatus	Deflocculating Agent Type	Concentration of Solution	Amount* (ml.)	Sample 1 Percent† of Particles Finer Than 0.005 mm.	Sample 1 0.001 mm.	Sample 2 0.005 mm.	Sample 2 0.001 mm.	Sample 3 0.005 mm.	Sample 3 0.001 mm.	Sample 4 0.005 mm.	Sample 4 0.001 mm.
S. D. T.	No deflocculating agent			58.2	38.6	54.9	34.6	Flocculated		Flocculated	
	Sodium Silicate	3° Baumé	20	63.1	51.1	58.6	45.9	53.7	40.8	56.9	29.8
	Sodium Pyrophosphate	0.5N	40	62.7	51.5	62.4	49.6	53.5	44.1	53.7	28.9
	Sodium Metaphosphate	0.5N	40	63.7	53.3	62.9	50.6	56.0	52.1	57.1	30.2
Mechanical Stirrer	No deflocculating agent			34.0	13.5	35.0	9.8	Flocculated		Flocculated	
	Sodium Silicate	3° Baumé	20	57.0	34.8	51.8	25.4	43.9	16.8	55.3	27.3
	Sodium Pyrophosphate	0.5N	40	59.6	48.4	59.5	48.2	54.2	42.7	53.3	28.4
	Sodium Metaphosphate	0.5N	40	62.8	51.0	59.9	48.0	54.8	43.0	56.3	29.6

Dispersion Apparatus	Deflocculating Agent Type	Concentration of Solution	Amount* (ml.)	Sample 5 Percent† of Particles Finer Than 0.005 mm.	Sample 5 0.001 mm.	Sample 6 0.005 mm.	Sample 6 0.001 mm.	Sample 7 0.005 mm.	Sample 7 0.001 mm.	Sample 8 0.005 mm.	Sample 8 0.001 mm.
S. D. T.	No deflocculating agent			Flocculated		15.7	6.0	Flocculated		Flocculated	
	Sodium Silicate	3° Baumé	20	28.8	7.1	17.8	10.4	4.8	2.7	F	F
	Sodium Pyrophosphate	0.5N	40	30.1	7.4	20.6	13.6	5.5	3.5	65.6	50.6
	Sodium Metaphosphate	0.5N	40	30.7	8.8	18.8	12.7	6.6	4.0	65.7	52.3
Mechanical Stirrer	No deflocculating agent			Flocculated		9.4	4.1	Flocculated		Flocculated	
	Sodium Silicate	3° Baumé	20	20.1	2.6	11.3	4.2	3.8	1.8	Flocculated	
	Sodium Pyrophosphate	0.5N	40	29.6	7.5	17.9	11.0	5.5	3.5	63.6	47.7
	Sodium Metaphosphate	0.5N	40	29.4	9.1	16.2	9.9	6.0	3.8	64.6	51.3

*Refers to the amount of deflocculating solution used in dispersing a sample of 50g. or 100g. into a one liter soil suspension.
†All percentages are the average of results from duplicate tests.

The degree of dispersion of a soil sample usually varies with the amount of deflocculating agent used. The trend of variation depends on the type of soil dispersed, the type of deflocculating agent used, and the apparatus and procedure of dispersion. Soils samples 1 and 6 were used to determine the relation between degree of dispersion and amount of each deflocculating solution. Both the mechanical stirrer and the S.D.T. were used in dispersing the soils.

The degree of dispersion obtained by different methods of dispersion can be compared by particle size measurements. This was followed in comparing the degree of dispersion obtained with different amounts of the

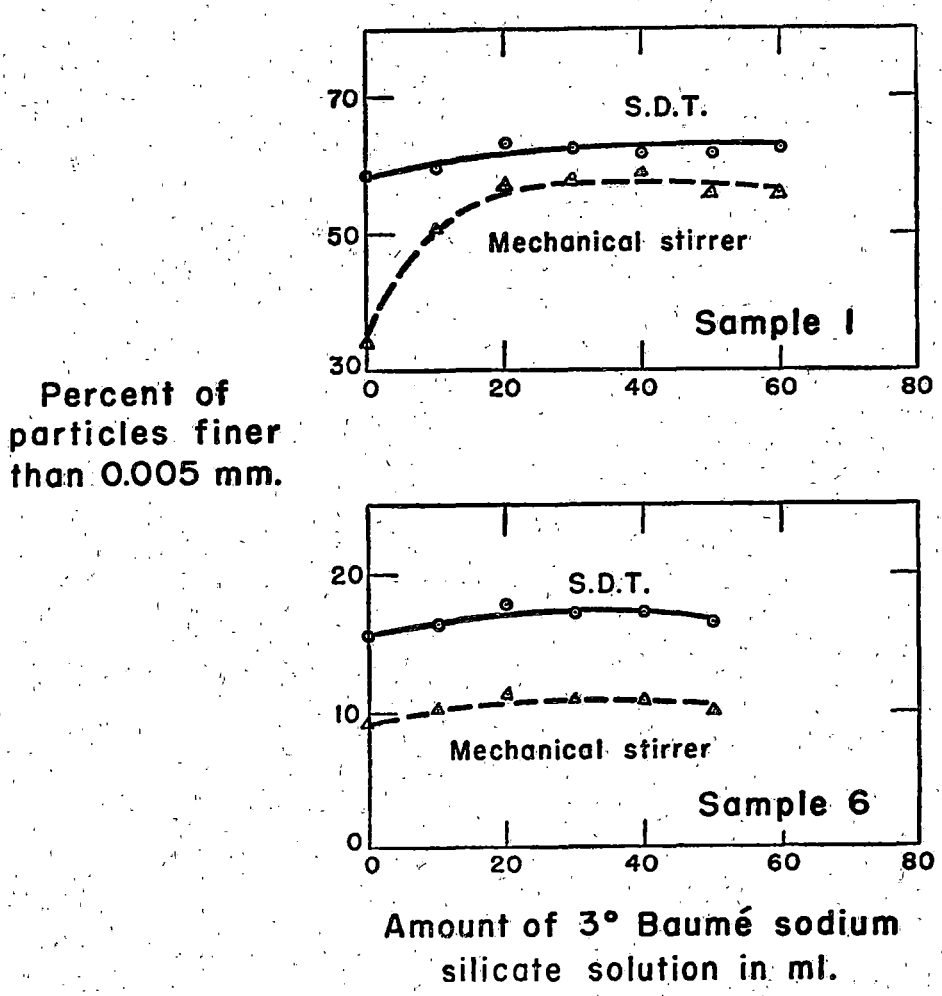

Fig. 1. Relation between amount of sodium silicate solution and degree of dispersion obtained with different dispersion apparatus.

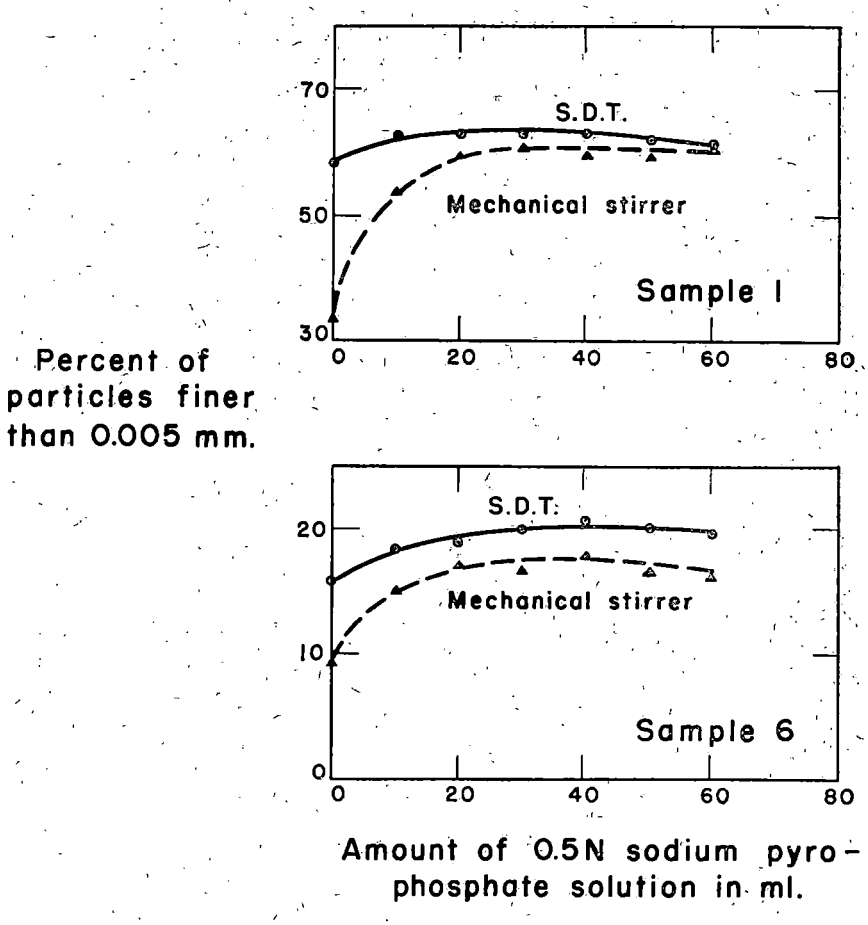

Fig. 2. Relation between amount of sodium pyrophosphate solution and degree of dispersion obtained with different dispersion apparatus.

three deflocculating solutions. The relations between the amount of each deflocculating solution and the degree of dispersion of the two samples are represented by the percent of particles finer than 0.005 mm. (figures 1, 2, 3). The curves for percentages finer than 0.001 mm. are similar. For equal amounts of solution, the S.D.T. gave a higher degree of dispersion than the mechanical stirrer and the amount of deflocculating agent used in the S.D.T. procedure was of less importance than in the mechanical stirrer procedure. For example the degree of dispersion of sample 1 varied only slightly with the amount of sodium metaphosphate solution when the soil was dispersed with the S.D.T. (figure 3). As a contrast, when dispersed with the mechanical stirrer, the degree of dispersion change substantially

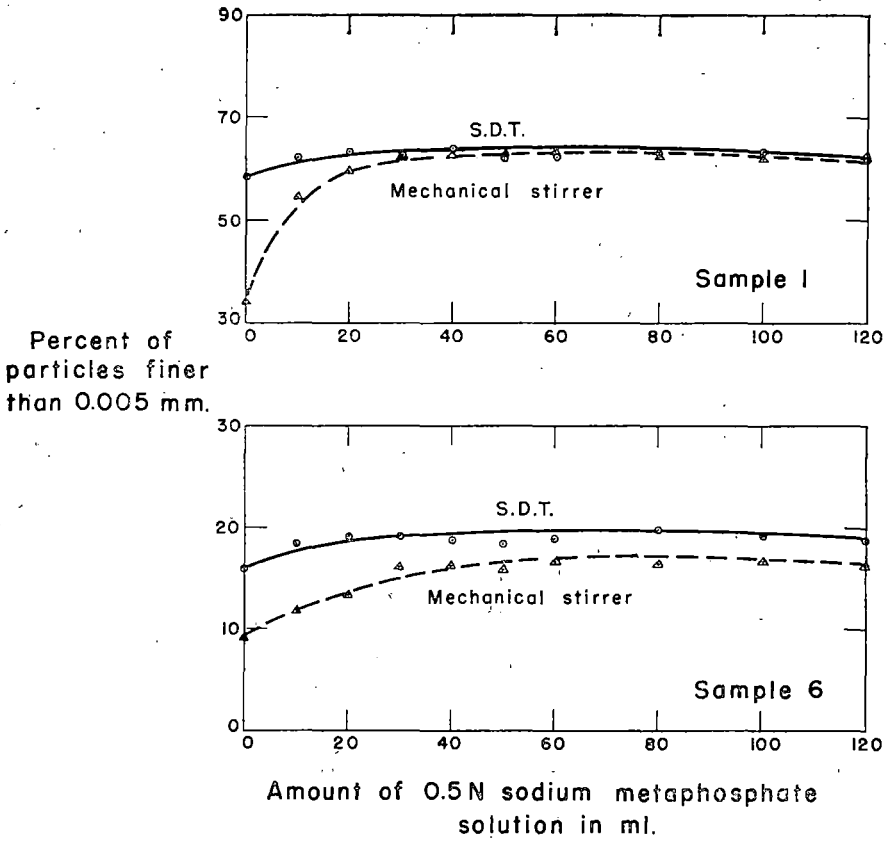

Fig. 3. Relation between amount of sodium metaphosphate solution and degree of dispersion obtained with different dispersion apparatus.

as the amount of sodium metaphosphate solution was varied from 10 to 30 ml. The data further indicate that, regardless of the type of dispersion apparatus used, the degree of dispersion of each sample remained practically unchanged when the amount of deflocculating solution used was about 20 ml. or more (ffgures 1, 2), or about 40 ml. or more (figures 3).

The three deflocculating agents were further compared in the dispersion of samples 2, 3, 4, 5, 7 and 8 with both types of dispersion apparatus specified in ASTM Method D422-51 and AASHO Method T88-49. The amounts of deflocculating solutions used for these samples were 20 ml. of sodium silicate solution as specified in ASTM Method D422-51 and AASHO Method T88-49, 40 ml. of sodium pyrophosphate solution, and 40 ml. of sodium metaphosphate solution (table III). With most of the samples, sodium silicate was found inferior to the other two deflocculating agents, and sodium metaphosphate gave slightly better results than sodium pyro-

phosphate. Therefore sodium metaphosphate was chosen for more detailed studies.

Comparison of Different Varieties of Sodium Metaphosphate

The sodium metaphosphate used in the foregoing experiments is one variety of the complex chemical also known as sodium hexametaphosphate or Graham's salt. The nomenclature of this group of chemicals is discussed in the appendix.

TABLE III. SOURCE AND STRUCTURE OF DIFFERENT VARIETIES OF SODIUM METAPHOSPHATE

Variety of Sodium Metaphosphate	Source	Structure*
A	Made by Calgon, Inc., Pittsburgh, Pa., and distributed under the trade name "Calgon"	Partially Microcrystalline
B	Made by Blockson Chemical Co., Joliet, Ill., available at Fisher Scientific Co., St. Louis, Mo., as sodium metaphosphate, C.P.	Glassy
C	Distributed by Fisher Scientific Co., St. Louis, Mo., as sodium hexametaphosphate	Crystalline
D	Prepared at Iowa Engr. Exp. Sta. Laboratory from sodium dihydrogen phosphate according to Tyner (5)	Glassy
E	Made by Rumford Chemical Works, Rumford, R. I., and sold under the trade name "Quadrafos"	Glassy
F	Made by Rumford Chemical Works, Rumford, R. I., and sold under the trade name "Metafos"	Glassy

*Based on examination with a petrographic microscope.

Since the different varieties of sodium metaphosphate sold by chemical supply companies may differ in their dispersing actions, experiments were made to compare six varieties (table III). Variety B was used in the previously discussed experiments to compare sodium metaphosphate with sodium silicate and sodium pyrophosphate. The source and structure or crystallinity of each variety are given. Only the glassy form of sodium metaphosphate has been suggested for use for soil dispersion purposes[7].

Soil samples 1 and 6 were chosen for the experiments. The S.D.T. was the only dispersion apparatus used. Since the amount of deflocculating solution needed for maximum dispersion may be different for different vari-

TABLE IV. COMPARISON OF EFFECTIVENESS OF DIFFERENT VARIETIES OF SODIUM METAPHOSPHATE IN SOIL DISPERSION

Deflocculating Agent (Sodium Metaphosphate) Variety	Amount* (ml.)	Sample No. 1 Percent of Particles Finer Than† 0.005 mm.	0.001 mm.	Sample No. 6 Percent of Particles Finer Than† 0.005 mm.	0.001 mm.
A	30	61.9	51.1	18.2	12.3
	40	63.1	52.1	19.0	13.3
	50	62.9	51.5	19.7	14.2
	60	63.6	51.3	18.9	13.5
B	30	62.4	51.3	19.1	11.7
	40	63.7	53.3	18.8	12.7
	50	61.7	52.0	18.4	11.9
	60	62.0	51.7	18.9	13.3
C	30	63.0	51.7	20.3	11.8
	40	62.6	52.2	18.9	12.2
	50	62.8	52.6	19.2	11.3
	60	62.9	52.1	18.2	11.7
D	30	62.5	50.8	20.3	11.2
	40	63.6	52.3	19.6	10.6
	50	62.8	51.4	19.3	10.6
	60	63.5	52.3	20.8	10.0
E	30	63.4	51.8	19.1	12.2
	40	63.0	51.6	18.7	14.3
	50	62.2	50.8	18.4	11.9
	60	64.4	53.2	18.4	12.8
F	30	62.6	51.5	18.1	15.0
	40	63.4	52.0	19.0	13.0
	50	61.5	51.6	18.5	13.4
	60	64.4	52.3	18.3	15.2

*Refers to the amount of 0.5N deflocculating solution used in dispersing a sample of 50 g. into a one liter soil suspension.
†All percentages are the average of results from duplicate tests.

eties of sodium metaphosphate, a number of solutions in the range of 30 to 60 ml. of 0.5N solution were tested to compare the six varieties (table IV).

The mechanical analysis results indicate that the degree of dispersion varies only slightly with the varieties and amounts of sodium metaphosphate tested. The type of structure of sodium metaphosphate appears to be of little consequence.

Because the mechanical analysis data for samples 1 and 6 showed no significant difference in the effectiveness of the six varieties of sodium metaphosphate, only two types, B and F, were used with the other six soil samples. Sodium metaphosphates, B and F, were selected mainly because of their comparative purity. The amounts of these two deflocculating solutions to be used with samples 2, 3, 4, 5, 7, and 8 were chosen on the basis of the data (figures 3 and 4). The curves representing dispersion with the S.D.T. show that when the amount of either sodium metaphosphate B or F solution is within the range 20 to 120 ml., the degree of dispersion is nearly

independent of the amount of deflocculating solution. For this reason, two amounts, 40 ml. and 100 ml., were used to cover this comparatively wide range. In these experiments only the S.D.T. dispersion apparatus was used.

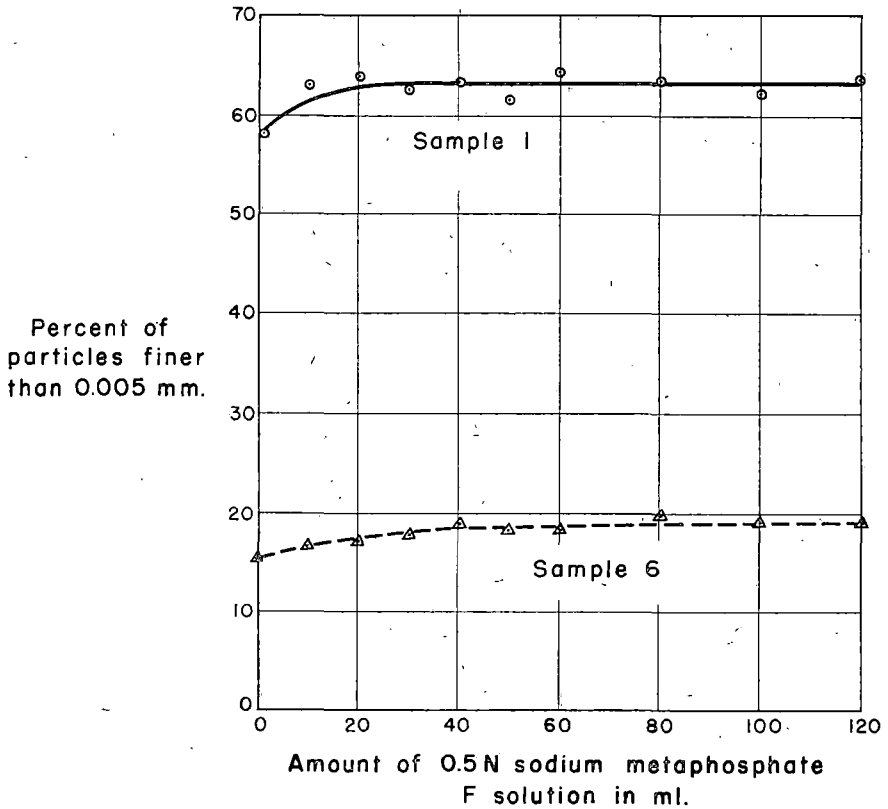

Fig. 4. Relation between amount of sodium metaphosphate F solution and degree of dispersion obtained with the S.D.T. apparatus.

Mechanical analysis data representing the degree of dispersion obtained with the two varieties of sodium metaphosphate indicate that sodium metaphosphate B and sodium metaphosphate F are equally effective (table V). An analysis of the data indicates that the use of 100 ml. of both kinds of sodium metaphosphate gives slightly better results than 40 ml.

Although all varieties of sodium metaphosphate were equally effective in dispersing the soils used in the preceding experiments, this might not be true with a greater variety of soils. Because of this, it seems desirable to recommend one kind of sodium metaphosphate in a standard method of mechanical analysis. Among sodium metaphosphates of equal effectiveness

TABLE V. COMPARISON OF TWO VARIETIES OF SODIUM METAPHOSPHATE IN SOIL DISPERSION

Deflocculating Agent (Sodium Metaphosphate)		Sample 1		Sample 2		Sample 3		Sample 4	
		Percent of Particles Finer Than†							
Variety	Amount* (ml.)	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.
B	40	63.7	53.3	62.9	50.6	56.0	52.1	57.1	30.2
	100	63.0	52.0	62.5	52.9	55.2	48.2	57.0	30.3
F	40	63.4	52.0	59.0	46.0	56.3	48.1	56.6	32.0
	100	62.2	53.1	60.8	50.3	55.7	51.0	57.8	30.5

Deflocculating Agent (Sodium Metaphosphate)		Sample 5		Sample 6		Sample 7		Sample 8	
		Percent of Particles Finer Than†							
Variety	Amount* (ml.)	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.	0.005 mm.	0.001 mm.
B	40	30.7	8.8	18.8	12.7	6.6	4.0	65.7	52.3
	100	30.2	10.3	19.1	12.5	6.5	4.3	66.2	54.0
F	40	30.8	8.3	19.0	13.0	7.4	4.0	67.1	52.1
	100	31.5	10.0	19.3	14.5	6.6	4.0	67.6	54.6

*Refers to the amount of 0.5N deflocculating solution used in dispersing a sample of 50 g. or 100 g. into one liter soil suspension.
†All percentages are the average of results from duplicate tests.

as deflocculating agents, preference should be given to a kind, such as sodium metaphosphate B, which is comparatively pure and readily available.

In a standard method of mechanical analysis, the recommendation of an amount of deflocculating solution that will give adequate dispersion to a great variety of soils is desirable. On the basis of the experiments reported herein, 100 ml. of 0.5N sodium metaphosphate B solution seems to be a safe amount to recommend.

Effect of Age of Sodium Metaphosphate B Solution on Its Dispersive Action

Sodium metaphosphate solutions may slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action[7]. Because of this possibility, experiments were made to determine the effect of aging on the dispersing action of 0.5N sodium metaphosphate B solution. The pH of the solution used was 6.8, and its temperature during storage was about 70 to 80° F. Both pH and temperature may affect the rate of reversion. Two amounts of this solution, 40 and 100 ml., were used in dispersing soil samples 1 and 6 with the S.D.T. apparatus. Results indicate that aging over a period of eight weeks had no appreciable effect on the dispersing action of the sodium metaphosphate B solution (table VI). Because of the limited extent of this experiment, no definite conclusion should be drawn. It appears safe to say, however, that aging of a solution up to one month will not decrease the dispersive action of sodium metaphosphate B.

TABLE VI. EFFECT OF VARIATION IN AGE OF SODIUM METAPHOSPHATE B SOLUTION ON ITS EFFECTIVENESS IN SOIL DISPERSION

Soil	Deflocculating Agent (Sodium Metaphosphate B) Amount* (ml.)	Age† (weeks)	Percent of Particles Finer Than‡ 0.005 mm.	0.001 mm.
Sample 1	40	0	63.7	53.3
		2	52.1	52.0
		4	63.0	52.0
		6	62.8	51.4
		8	62.6	51.0
	100	0	63.0	52.0
		2	62.2	51.5
		4	62.7	53.1
		6	64.3	52.5
		8	62.3	51.3
Sample 6	40	0	18.8	12.7
		2	20.2	13.8
		4	18.4	14.2
		6	20.4	13.3
		8	19.5	13.1
	100	0	19.1	12.5
		2	19.7	14.3
		4	18.9	15.0
		6	20.0	13.9
		8	18.9	13.5

*Refers to the amount of 0.5N deflocculating solution used in dispersing a 50 g. sample into a one liter soil suspension.

†Age refers to the time period after the solution is prepared.

‡All percentages are the average of results from duplicate tests.

SECONDARY EFFECTS OF A DEFLOCCULATING AGENT ON MECHANICAL ANALYSIS RESULTS

In addition to affecting the degree of dispersion of a soil sample, a deflocculating agent may have other important effects on mechanical analysis results. These effects, which will be referred to as secondary effects, include the changes in the specific gravity of the soil particles and in the viscosity and specific gravity of the suspending medium.

In the standard methods of mechanical analysis, ASTM Method D422-51, AASHO Method T88-49, the percentage and the diameter of soil particles remaining in suspension after a given sedimentation period are computed by the following equations. Equation 1 is for tests using Bouyoucos hydrometer. When specific gravity hydrometers are used, the equation for computing the percentage of soil in suspension will be slightly different, but the same variables are involved.

$$P = \frac{Ra}{W} \times 100 \qquad (1)$$

Where:

P = percentage of originally dispersed soil remaining in suspension.
R = hydrometer reading (temperature correction should be applied if necessary).
W = weight in grams of soil originally dispersed minus the hygroscopic moisture.
a = constant depending on the specific gravity of soil dispersed and the specific gravity of the suspending medium.

$$d = \frac{30\ nL}{980\ (G - G_1)\ T} \qquad (2)$$

Where:

d = maximum particle diameter in mm.
n = coefficient of viscosity of the suspending medium in poises.
L = distance in cm. through which soil particles settle in a given period of time.
T = time in minutes, period of sedimentation.
G = specific gravity of soil particles.
G_1 = specific gravity of the suspending medium.

In computing the percentage and the diameter of soil particles remaining in suspension from equations (1) and (2), it is usually assumed that values of *R* and *a* in equation (1) and of *n*, *G*, and G_1 in equation (2) are not significantly affected by the use of a deflocculating agent. Actually these values may be substantially affected, especially when a relatively large quantity of deflocculating solution is used.

These experiments were conducted to determine the secondary effects resulting from the use of sodium metaphosphate B. In these experiments the S.D.T. dispersion apparatus was used for samples 1 and 6.

Effect on Specific Gravity of Soil Dispersed

In experiments with homoionic soils, the specific gravity of a soil was found to vary with the kind of adsorbed cation[9]. Since exchange of cations, as well as other chemical changes, may take place when a deflocculating agent is added to a soil water mixture, there is a possibility for a change to occur in the specific gravity of the soil particles.

The exchangeable cations in sample 6 are mainly sodium, potassium, and calcium, with the latter cation occupying about 80% of the exchange positions. Those in sample 1 were not determined analytically but are estimated to be mainly hydrogen. The cation exchange capacities of samples 6 and 1 are 40.0 and 13.4 m.e./100g. respectively.

Experiments to determine the effect of sodium metaphosphate B on the specific gravity of samples 1 and 6 consisted of specific gravity measurements before and after dispersion using ASTM Standard Method D854-45T. Following dispersion with 100 ml. of 0.5N solution, the soil suspension was

left undisturbed for 24 hours, filtered, washed with distilled water, and then dried. Results indicate that no significant change in specific gravity occurred (table VII).

TABLE VII. COMPARISON OF SPECIFIC GRAVITIES OF SOILS BEFORE AND AFTER DISPERSION WITH 100 ML. 0.5N. SODIUM METAPHOSPHATE B SOLUTION

Sample No.	Specific Gravity*, 20C/20C	
	Before dispersion	After dispersion
1	2.714	2.711
6	2.729	2.724

*Specific gravity values are the average of the data from triplicate tests.

Effect on Viscosity of Suspending Medium

The relation between the viscosity of the suspending medium and the diameter of soil particles in suspension is shown by equation (2). In the AASHO and ASTM standard methods of mechanical analysis, the viscosity of distilled water is taken as the viscosity of the suspending medium. Actually, when a deflocculating agent is used, the suspending medium will be a combination of water and the deflocculating solution; and the viscosity of the resulting suspending medium may be appreciably different from that of water. This is illustrated by the viscosity measurements of liquids A, B, C, and D representing different kinds of suspending medium (table VIII). A Cannon-Fenske-Ostwald type viscometer was used, and the test procedure recommended in ASTM Method D445-46T was followed. The viscosity of liquid D is about 3.4 percent higher than that of distilled water (see liquid A). Such a difference in viscosity will result in a relative difference of about 1.7 percent in the diameter of soil particles computed by equation (2).

Theoretically, neither the viscosity of water nor that of water mixed with a deflocculating agent should be used as n in equation (2). Consider a soil suspension containing silt size and clay size particles. Since the silt size material settles much faster than the clay size, especially the sizes finer than 0.001 mm., the medium through which silt size material settles is a soil suspension comprised of clay size material, deflocculating agent, and water. The viscosities of soil suspensions prepared from the fraction finer than 0.001 mm. in samples 1 and 6 may be as much as 27 percent higher than that of pure water. In using equation (2), this would result in a difference of about 13 percent in the diameter of soil particles.

From the somewhat limited experimental results discussed above, it appears that the viscosity value used in computing particle diameters for an accurate mechanical analysis should be as nearly as possible that of the actual suspending medium. The most practical approach in routine tests might be to apply corrections to the diameters as computed in the conven-

tional manner. The correction will vary not only with the temperature of the soil suspension but also with the particle size composition of the soil sample and the value of the particle diameter. One way to obtain the correction values for routine testing purposes is to arbitrarily divide the common types of soil into several groups and to determine the corrections needed for the different particle size ranges in each group. The temperature correction can either be included in these correction values, or it can be applied separately.

TABLE VIII. COMPARISON OF VISCOSITIES OF DISTILLED WATER, DISTILLED WATER WITH DEFLOCCULATING AGENTS, AND SOIL SUSPENSIONS.

	Liquid	Viscosity at 68° F. (centipoise)
A	(Distilled water)	1.004
B	(20 ml. 3° Baumé sodium silicate solution mixed with 961 ml. distilled water)	1.018
C	(40 ml. 0.5N sodium metaphosphate B solution mixed with 941 ml. distilled water)	1.025
D	(100 ml. 0.5N sodium metaphosphate B solution mixed with 881 ml. distilled water)	1.038
Soil suspension* prepared from fraction finer than 0.001 mm. in sample 1 with the following suspending medium:		
	Liquid A	1.026
	Liquid B	1.031
	Liquid C	1.056
	Liquid D	1.097
Soil suspension* prepared from fraction finer than 0.001 mm. in Sample 6 with the following suspending medium:		
	Liquid A	1.116
	Liquid B	1.195
	Liquid C	1.251
	Liquid D	1.274

*The soil suspension prepared from the fraction finer than 0.001 mm. in Sample 1 contains about 20 g. in 1000 ml. suspension; that prepared from the fraction finer than 0.001 mm. in Sample 6 contains about 3 g. in 1000 ml. suspension.

Effect on Specific Gravity of Suspending Medium

The addition of a deflocculating agent to a soil water mixture will change the density or specific gravity of the suspending medium, which will affect particle size determinations in two ways. The value of a in equation (1) and that of G_1 in equation (2) may be significantly affected. The hydrometer reading R in equation (1) is influenced in the following manner.

Regardless of the type of hydrometer used, hydrometer readings give the difference between the specific gravity of the soil suspension and that of water. If the suspending medium is water only, the hydrometer reading R represents the increase in specific gravity due to the presence of suspended soil particles. If water mixed with a deflocculating agent is the suspending medium, the hydrometer reading R represents the increase in the specific gravity due to the presence of both suspended soil particles. If water mixed with a deflocculating agent is the suspending medium, the hydrometer reading R represents the increase in the specific gravity due to the presence of both the suspended soil particles and the deflocculating agent.

The change in the values of a and G_1 can be determined by measuring the specific gravity of the actual suspending medium, water containing a deflocculating agent. For example, when 100 ml., of 0.5N sodium metaphosphate B solution is contained in one liter of soil suspension, the specific gravity of the suspending medium at 67° F. will be about 1.003, which is approximately 0.5 percent higher than the specific gravity of water at the same temperature. A difference of this amount will result only in a change of about 0.3 percent in the percentage values and of about 0.2 percent in the diameter values obtained from equations (1) and (2) respectively. These small changes can probably be overlooked in routine mechanical analyses.

The idea of correcting the hydrometer reading R for the presence of a deflocculating agent is not new. Many laboratories apply such a correction when the quantity of deflocculating solution used is relatively large.

Hydrometer readings may be corrected by subtracting the hydrometer reading of the suspending medium (water plus a deflocculating agent) from the reading taken in the soil suspension. (With specific gravity hydrometers, only the decimal portion of the hydrometer reading will be subtracted.) The hydrometer reading of the suspending medium can be determined by a hydrometer measurement in water containing the amount of deflocculating agent in the soil suspension. The correction constant can be determined from the hydrometer reading (with the Bouyoucos hydrometer, the reading is the constant).

When different amounts of a given deflocculating solution are being investigated, the following equations may simplify the determination of correction constants:

For Bouyoucos hydrometer

$$C = \frac{mR_d}{1000 - \frac{W}{G}} \tag{3}$$

For specific gravity hydrometer

$$C = \frac{m(R_d - 1)}{1000 - \frac{W}{G}} \tag{4}$$

Where:

C = correction constant.

m = amount of deflocculating solution in ml. contained in one liter of soil suspension.

R_d = hydrometer reading of deflocculating solution at specified temperature.

W = weight in grams of soil originally dispersed minus the hygroscopic moisture.

G = specific gravity of soil dispersed.

In deriving equations (3) and (4), the Bouyoucos and the specific gravity hydrometer readings for water at the specified temperature (usually 67 or 68° F.) are assumed to be zero and one respectively. For practical applications of the two equations, the specific gravity G can be assumed as 2.65 because it has little effect on the computed correction constant. It should also be mentioned that the correction constant computed from either equation (3) or (4) is always positive in value and should be subtracted from hydrometer readings taken in the soil suspension.

This method of determining correction constants is valid only if it can be assumed that chemical changes caused by the addition of a deflocculating agent to a soil water mixture do not significantly affect hydrometer readings taken in the soil suspension.

A direct test of the validity of this assumption is to compare the correction constant determined by equations (3) or (4) with the required correction as determined experimentally. The required correction equals the difference between the hydrometer reading taken in a soil suspension containing a deflocculating agent and that taken in a similar soil suspension without the deflocculating agent. Since the degree of dispersion of a soil sample may be greatly affected by the use of a deflocculating agent, the experimental determination of the required correction must be accomplished in such a way that any change in the degree of dispersion of the soil sample will not significantly affect hydrometer readings taken in the soil suspension. This can be done by using clay size soil samples. A discussion of two such determinations follows.

The clay size material was collected by the layer method[2] from soil samples 1 and 6. The clay separated from sample 1 was finer than 0.0005 mm. in size, and that from sample 6 was finer than 0.005 mm. Samples of each separated clay material were soaked for over 18 hours in either distilled water or distilled water containing sodium metaphosphate B (table IX) before being dispersed with the S.D.T. for the hydrometer test. The hydrometer tests were conducted in essentially the same manner specified in the AASHO and ASTM standard methods of mechanical analysis.

As mentioned, the purpose of using such clay size material was to eliminate the degree of dispersion variable from the tests. Thus, any variation in the degree of dispersion of a sample consisting of particles finer than

0.0005 mm. will not significantly affect hydrometer readings taken within one hour after the beginning of sedimentation. Similarly, hydrometer readings in a soil suspension consisting of minus 0.005 mm. material will not be significantly affected by a variation in the degree of dispersion, if the readings are taken within 15 minutes after the beginning of sedimentation.

TABLE IX. COMPARISON OF EXPERIMENTAL AND COMPUTED CORRECTION CONSTANTS FOR HYDROMETER READINGS

Soil	Deflocculating Agent		Hydrometer Reading of Soil Suspension at the elapsed time indicated†		Difference in hydrometer readings of soil suspension with and without deflocculating agent‡		Correction constant determined by equation (3)
	Type	Amount* (ml.)	15 min.	60 min.	15 min.	60 min.	
Sample 1, fraction finer than 0.0005 mm.	No deflocculating agent		14.5	14.5	0	0	....
	Sodium metaphosphate B	40	17.0	17.0	2.5	2.5	2.5
		100	21.0	21.0	6.5	6.5	6.4
Sample 6, fraction finer than 0.005 mm.	No deflocculating agent		6.5	Not taken	0	....	....
	Sodium metaphosphate B	40	9.0	Not taken	2.5	....	2.5
		100	13.0	Not taken	6.5	....	6.4

*Refers to the amount of 0.5N deflocculating solution used in preparing one liter of soil suspension.

†Bouyoucos type hydrometer was used in all tests. The temperature of soil suspension was maintained at 67° F. during the hydrometer test.

‡Example: Hydrometer reading of suspension without deflocculating agent = 14.5, hydrometer reading of suspension with 40 ml. deflocculating solution = 17.0, difference in hydrometer readings = 17.0 — 14.5 = 2.5.

Carbonates in a soil sample may influence chemical changes taking place in a soil suspension in which sodium metaphosphate is the deflocculating agent. The minus 0.0005 mm. clay material used in the tests contained a negligible amount of carbonates; the 0.005 mm. clay contained about eight percent of carbonates, principally calcium carbonate.

Hydrometer readings of the prepared soil suspensions taken at different sedimentation times are given (table IX), but those taken before 15 minutes are not shown, since they were the same as those taken at 15 minutes due to the smallness of the particle sizes contained in the suspensions. To eliminate the effect of variation in degree of dispersion on hydrometer readings, no readings were taken after 60 minutes of sedimentation in the soil suspensions prepared with minus 0.0005 mm. material and none after 15 minutes in suspensions prepared with the minus 0.005 mm. material.

The required correction constant for each suspension containing sodium metaphosphate B, obtained by subtracting the hydrometer reading of the suspension from the hydrometer reading of a similar suspension containing no deflocculating agent, is given together with the correction constants computed by equation (3). Considering that hydrometer readings were taken to the nearest half division, the required and the computed corrections are practically in complete agreement. According to this experiment, the assumption made in developing equations (3) or (4) seems valid.

CONCLUSIONS

1. Among the three chemicals compared, sodium metaphosphate is the most promising deflocculating agent.
2. Among the different varieties of sodium metaphosphate compared, variety B appears to be well suited for adoption as a deflocculating agent in standard methods of mechanical analysis.
3. On the basis of the results with the soils tested, the use of 100 ml. of 0.5N sodium metaphosphate B solution in making one liter of soil suspension is recommended.
4. To avoid a possible decrease in its dispersive action, it seems advisable to make up fresh sodium metaphosphate B solutions about every 30 days.
5. The effect of sodium metaphosphate B on the specific gravity of soils tested was found to be insignificant.
6. The viscosity of water is used as that of the suspending medium in the present standard methods of mechanical analysis in computing particle diameters. A correction should be applied to the computed diameter for accurate mechanical analysis results to compensate for the difference between the viscosity of water and that of the actual suspending medium.
7. The suggested method for determining the correction constant to compensate for the change in specific gravity of the suspending medium due to the use of a deflocculating agent appears to be valid.

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APPENDIX

Nomenclature of Sodium Polyphosphates

No nomenclature for the sodium polyphosphates has yet been adopted. The following is one method of classifying the different sodium polyphosphates.

The sodium polyphosphates may be classified according to their structure into two groups, the crystals and the glasses. The crystalline sodium polyphosphates include sodium metaphosphate, sodium pyrophosphate, and sodium tripolyphosphate. The glassy sodium polyphospates include sodium tetraphosphate, sodium hexametaphosphate, and also sodium metaphosphate. The two names, sodium hexametaphosphate and sodium metaphosphate, are often used interchangeably. Manufacturers use trade names such as "Calgon," "sodium Polyphos," "Quadrafos," and "Metaphos" in referring to their glassy sodium polyphosphate products.

As mentioned, sodium metaphosphate can be either crystalline or glassy. Different varieties of this chemical are available from chemical supply

companies. The complicated properties of this group of chemicals have been identified as follows[9, p. 740]:

> Metaphosphoric acid, HPO_3, and its salts possess the most complicated properties of all the acids of phosphorus; because, on the one hand, they have a strong tendency to polymerize, when varied products of high molecular weight can be formed; while, on the other hand, these products show isomerism through different arrangements within the individual molecules, so that varying constitutions may appear with the same molecular complexity. The relations of the metaphosphates are still so confused, in spite of numerous investigations, that the practice of giving definite formulae for the products must be given up for the present.

MATHEMATICAL ANALYSIS OF A LAYER EXTRACTION METHOD FOR SEPARATING CLAY-SIZE MATERIAL FROM SOILS

by

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(Clays and Clay Minerals, 462-479. 1954.)

ABSTRACT

A layer extraction method has been developed for obtaining a representative sample of clay-size material from a soil suspension. This method is especially advantageous in preparing a sample of relatively large size, and the procedure is simpler than that of the decantation method, a comparable method of extraction.

In the layer method, the position and the thickness of the layer to be extracted from a soil suspension can be varied, and one or more extractions can be made. The effect of these variables on the size composition and the quantity of the extracted material is analyzed mathematically. Based upon such an analysis, the proper position and thickness of the layer and the desirable number of extractions can be determined to suit the requirements of a particular test.

INTRODUCTION

Properties of common soils depend to a large extent upon the amount and characteristics of the clay-size material contained in the soils. For this reason, it is often desirable to separate the clay-size material from a natural soil and to study the characteristics of the material separated.

Whereas different types of elutriators[1, 5] have been used for making the required separation, sedimentation methods are in common use, especially for separating materials finer than 0.002 mm. The procedure for making the separation by sedimentation methods includes dispersion of a soil sample[2, 3, 10], sedimentation of the suspended soil particles by gravity or by centrifugation, extraction of the required clay-size material from the soil suspension[6, 7, 8, 9] and drying, if necessary, of the extracted soil suspension. This paper presents the theory of a layer method of extraction.

While this method was developed primarily for separating the clay-size material from soils, it can be used for separating any size fraction from any material whose component parts can be differentiated by sedimentation

methods. Various types of apparatus may be used in the layer extraction method. Experiments with extraction apparatus are expected to be made in the future.

The method of extraction used has an important bearing on the size composition of the clay-size material separated from a soil sample. Usually it is desirable that the gradation of the material collected be the same as the gradation of the clay-size material in the dispersed sample. The extraction procedure should also be relatively simple. These two requirements can be used as criteria in evaluating different extraction methods.

Decantation Method

The method herein referred to as the *decantation method* is commonly used when a comparatively large quantity of clay-size material is needed. The procedure is as follows:

A soil sample is dispersed in distilled water and the resulting suspension allowed to settle in a container. After a given period of sedimentation, the portion of soil suspension containing soil particles smaller than a specified size is removed from the container by decantation or siphoning. The container is then refilled with distilled water to the original level, the diluted soil suspension allowed to settle after shaking, and the extraction process repeated. This procedure is continued until the portion to be removed contains little or no suspended soil particles. The several withdrawals are combined to obtain a representative sample of the desired clay fraction.

The decantation method is satisfactory when the proper number of decantations are made; if not, the gradation of the material collected will not be representative. Perhaps the main disadvantage of this method is that it is exceedingly time-consuming, both as to the large number of repeated decantations required (possibly 20 to 30 with fine-grained soils) and to the quantity of water that must be separated from the clay material by drying or centrifuging. Another disadvantage is that the repeated decantations tend to magnify experimental errors.

Other Methods

A pipette may be used for extraction purposes[4]. After a given sedimentation period, the pipette is lowered into the soil suspension to a predetermined depth and a small quantity, usually 5 to 25 ml., is removed with the pipette. Although a representative sample can be obtained in this manner, the use of the pipette is limited to experiments requiring only a small amount of clay-size material.

The extraction of clay-size material from a soil suspension can be accomplished with a continuous-type supercentrifuge. The supercentrifuge serves to accelerate sedimentation as well as to separate the desired clay fraction. This method is especially useful for separating clay material into

different particle-size range fractions. The use of the supercentrifuge has been discussed[1].

The principle of the supercentrifuge method is similar in many respects to that of the decantation method. While long sedimentation periods can be eliminated by use of the supercentrifuge, the repeated treatments necessary for complete separation and the necessity for collecting the clay-size material from a large amount of suspension make the method time-consuming. Except for those laboratories which do frequent testing, the expensive and comparatively complex equipment requirements are a decided disadvantage in using the supercentrifuge method.

LAYER EXTRACTION METHOD

The layer extraction method was developed for the purpose of obtaining representative samples of the material finer than a given size by a simple and comparatively short procedure. This method is suitable for separating relatively large quantities of clay-size material from soils. Although the layer method can be used to fractionate further the clay-size material separated, it probably is advantageous to use a supercentrifuge.

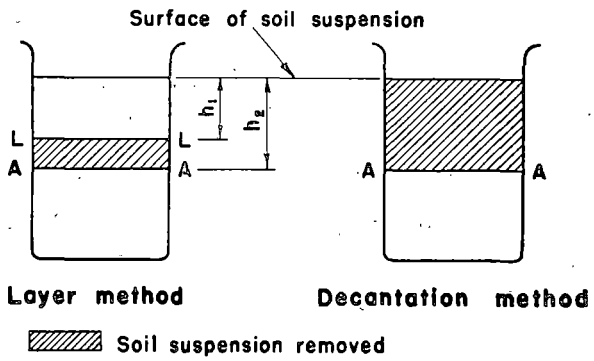

Fig. 1. Soil suspension removed by layer method and decantation method of extraction.

Among the different extraction methods, the decantation method is perhaps most comparable with the layer method (figure 1). Level A-A in figure 1 represents the level above which no particles coarser than the maximum particle size of the desired clay fraction will remain in suspension after a given sedimentation period. In the decantation method of extraction, the soil suspension above level A-A is removed. The portion of soil suspension removed in the layer method is a layer bordering or embracing level A-A. When the layer method is used, repeated removals are not required to obtain a representative sample; but if the amount of material

collected from the first extraction is insufficient, additional extractions from the remaining soil suspension can be made. After the first extraction, the soil suspension remaining in the container is composed of the portion below level A-A and the portion above level L-L. This remaining suspension can be reshaken and another layer removed after sedimentation.

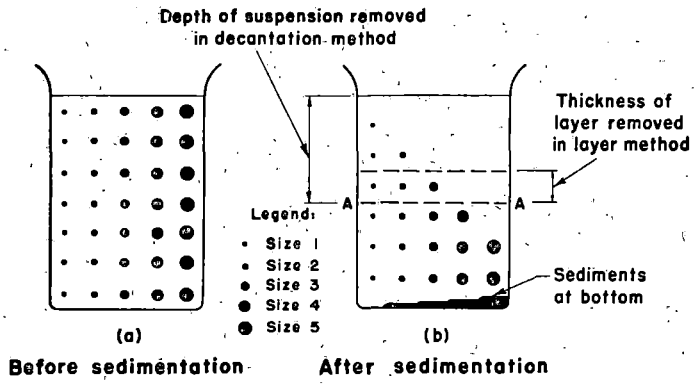

Fig. 2. Cross sections of a hypothetical suspension before and after sedimentation.

The diagrams in figure 2 illustrate the basic theory of the layer method. Only five particle sizes are contained in the dispersed hypothetical sample. The distribution of the five particle sizes in the suspension is shown schematically in the diagrams, which show that all particles are uniformly dispersed throughout the entire depth of the suspension before settling begins.

If size 3 is the maximum size in the desired fraction, the ratio of the three sizes contained in the fraction is 1:1:1. The distribution of the suspended particles after a given period of sedimentation and the position of the layer to be extracted by the layer method are shown in figure 2(b). Note that the ratio of particle sizes 1, 2, and 3 in the layer is also 1:1:1. A representative sample can thus be obtained from a single extraction.

Mathematical Analysis

The theory of the layer method of extraction can be verified either by laboratory experiments or by mathematical analysis. The latter method is used in this presentation. To avoid complex mathematical expressions, the analysis is presented by numerical examples. All equations used in the numerical examples are derived in Appendix I, and their application in computing the theoretical gradation of materials obtained is demonstrated in Appendix II. In these computations the material collected from an extracted layer is assumed to be the same as the material contained in the layer prior to extraction.

TABLE I. GRADATION OF HYPOTHETICAL SOIL

Particle Size, mm.	Percent Finer: On the basis of the whole sample	Percent Finer: On the basis of the fraction finer than 0.002 mm.
1.00	100.0	
0.074	96.0	
0.005	53.0	
0.0025	43.1	
0.00225	42.0	
0.00200	40.0	100.0
0.00175	38.2	95.5
0.00150	36.2	90.4
0.00100	30.7	76.9
0.00060	23.9	59.8
0.00030	14.7	36.7
0.00010	0	0

A hypothetical soil with gradation as shown in table I, and figure 3 was used in the numerical examples. In separating the clay-size material from this soil, it is assumed that a 150 g. sample is dispersed in distilled water and the resulting suspension put in a container, shaken, and allowed to settle by gravity. It is further assumed that the depth of the suspension in the container is 21.00 cm.

If the material finer than 0.00200 mm. is the clay fraction desired, computations can be made to determine level A-A (figure 1) above which no particles coarser than 0.00200 mm. will remain in suspension after a given period of sedimentation. According to Stokes' law[1],

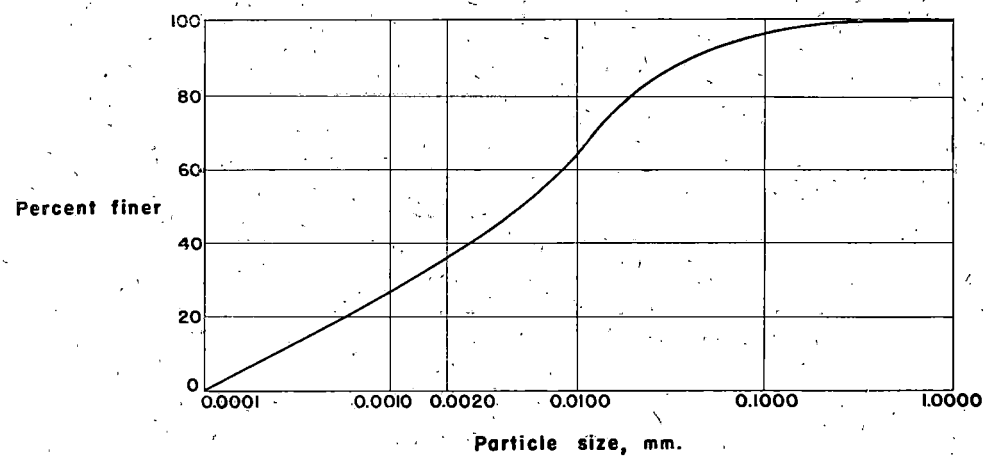

Fig. 3. Particle-size accumulation curve of hypothetical soil.

$$h = \frac{980\ (G-G_i)\ TD^2}{30\ n} \qquad (1)$$

where h = distance in centimeters through which soil particles settle
G = specific gravity of soil particles
G_i = Specific gravity of the suspending medium, in this case water
T = time in minutes, period of sedimentation
D = particle diameter in millimeters
n = absolute viscosity of the suspending medium in poises

If $G = 2.730$
$G_i = 0.998$ (water at 67° F.)
$T = .360$ min.
$n = 0.0102$ poises (water at 67° F.)

then $h = 2.0 \times 10^6\ D^2$ (2)

In this case $D = 0.002$ mm.
and from equation (2) $h = 8.00$ cm.

Level A-A is therefore 8.00 cm. below the surface of the soil suspension.

Among the assumptions above, those relating to the method of sedimentation and to the gradation of the hypothetical soil should be further clarified. Although gravity sedimentation is assumed in the numerical examples, the layer method can be used as well for extracting clay-size material from a soil suspension in which the sedimentation is accelerated by centrifugation. The gradation of the hypothetical soil contains no material finer than 0.00010 mm. and the segment of the particle-size accumulation curve representing the fraction finer than about 0.0025 mm. is nearly a straight line (figure 3). Actually a soil having almost any gradation could have been selected as the hypothetical soil for the mathematical analysis,

TABLE II. SPECIAL CASES OF THE LAYER METHOD

	Position of the layer with respect to Level A-A*	Top of the Layer h_1*, cm. (Distance from surface of soil suspension)	Top of the Layer Limiting Particle Size, mm.	Bottom of the Layer h_2*, cm. (Distance from surface of soil suspension)	Bottom of the Layer Limiting Particle Size, mm.	Thickness of the layer, cm.
Case I	Above level A-A	4.50	0.00150	8.00	0.00200	3.50
Case II	Below level A-A	8.00	0.00200	12.50	0.00250	4.50
Case III	Partly above and partly below level A-A	6.12	0.00175	10.12	0.00225	4.00
Case IV	Same position as case III	4.50	0.00150	12.50	0.00250	8.00

*See figure 1.

but the analysis is greatly simplified by using the hypothetical soil. In general, conclusions drawn from the analysis will hold true for all soils.

In the layer method of extraction, the position and the thickness of the layer to be extracted from a soil suspension can be varied. In addition, one or more extractions can be made to obtain a sample of the desired clay fraction. These variables will be considered in the verification of the layer method theory.

Four typical cases representing possible variations in the position and thickness of the layer are given in table II. The term *limiting particle size* used in the table refers to the maximum particle size occurring at a specific level in the soil suspension after a given period of sedimentation.

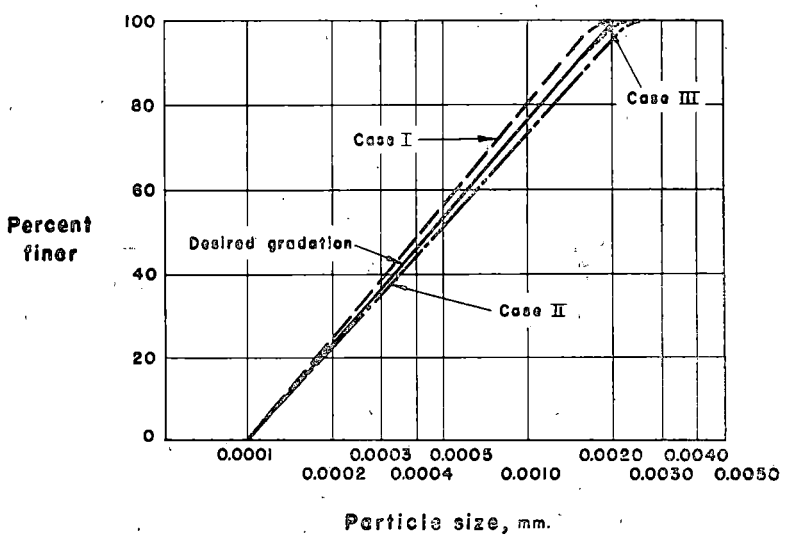

Fig. 4. Particle-size accumulation curves showing differences in gradation of clay-size material obtained from layers positioned as in case I, II, and III (table II).

Position of Layer

The gradation of the clay-size materials obtained from layers positioned as in cases I, II, and III can be determined from equations presented in Appendix I. A computation which illustrates this is given in Appendix II. Computed gradations for the three cases are shown as accumulation curves in figure 4. The gradation in case III is closest to the desired gradation, but the material obtained from this layer contains a small amount of particles coarser than 0.002 mm., the maximum particle size of the desired clay fraction. The term *desired gradation* refers to the gradation of the desired clay fraction, in this case, the minus 0.002 mm. material. Case I material,

on the other hand, meets the maximum particle-size requirement but is not as satisfactory with respect to overall gradation. Because both the thickness of the extracted layer and the method of extraction affect the gradation, it is impossible to select a layer position which will give the best results without consideration of them.

Thickness of Layer

If the position of the layer is properly chosen, the thinner the layer the more closely will the gradation of the material obtained approach the desired gradation. This is illustrated by comparing cases III and IV (table II). Figure 5 shows the desired gradation as well as the computed gradations of materials obtained from layers positioned as in cases III and IV. Note that the material from the thinner layer, case III, has the more desirable gradation.

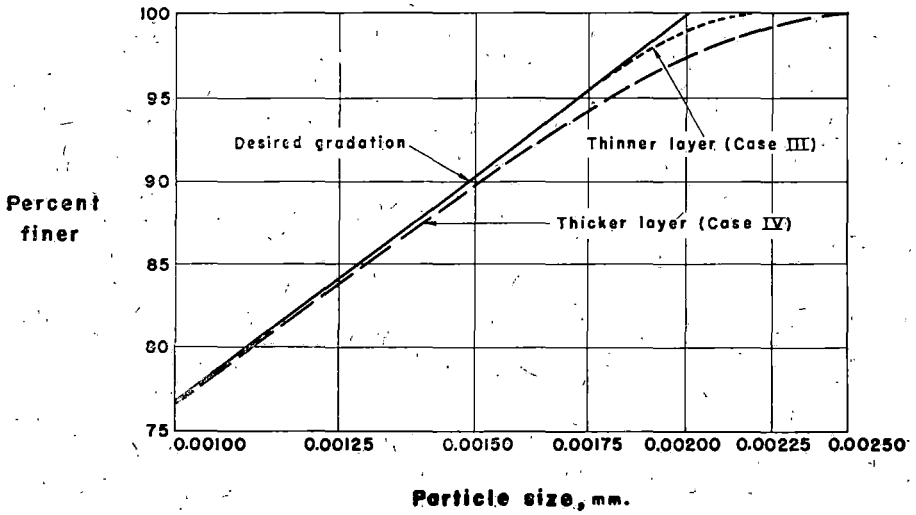

Fig. 5. Upper portion of particle-size accumulation curves showing the difference in gradation of clay-size material obtained from layers of various thicknesses.

In practical application of the layer method, the choice of layer thickness would also be influenced by the quantity of clay-size material desired. If thinner layers are chosen, more time and effort will be necessary to obtain a sample of the desired quantity.

Number of Extractions

Although repeated extractions are not required in using the layer method, three or four extractions from a soil suspension may be necessary to obtain a sample of larger size. The difference in gradation of the materials

obtained from repeated extractions will be very small and perhaps can be considered within the range of experimental error. This is illustrated by the following comparison of the gradations of the clay-size material obtained from the first and second extractions of a layer positioned as in case I.

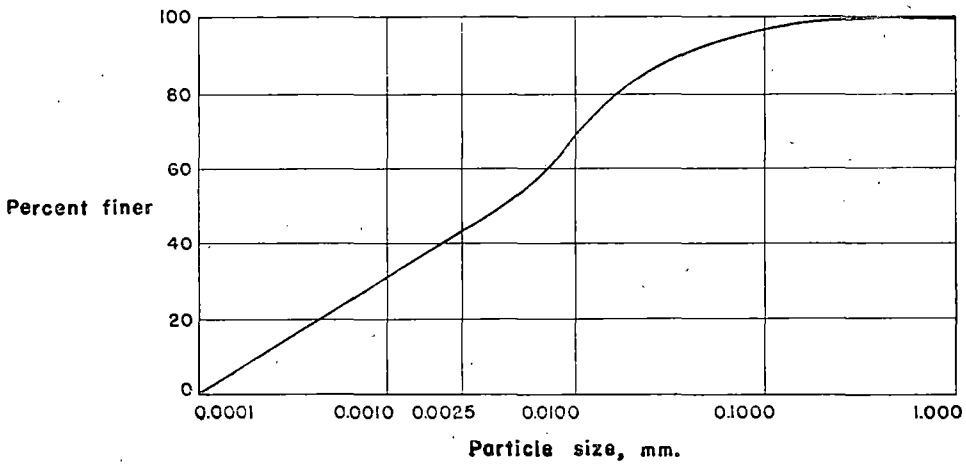

Fig. 6. Particle-size accumulation curve of soil in the suspension after first extraction.

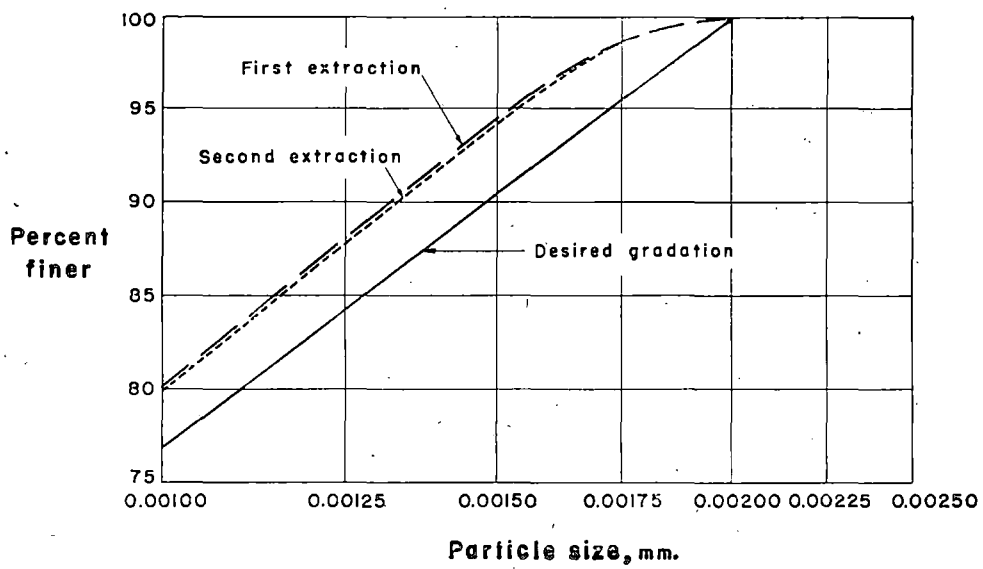

Fig. 7. Upper portion of particle-size accumulation curves showing differences in gradation of clay-size material in first and second extractions from case I layer (table II).

Since the original depth of the hypothetical soil suspension was assumed to be 21.00 cm., the removal of the first 3.50 cm. layer (see table II) will leave 17.50 cm. of suspension in the container. This remaining suspension is re-shaken, and after a given sedimentation period another 3.50 cm. layer is withdrawn. By using the theoretical particle-size accumulation curve of the soil in the suspension after the first extraction (figure 6) and the equations in Appendix I, the gradation of the material from the second extraction can be determined.

Portions of the computed particle-size accumulation curves representing the clay-size material obtained from the two extractions are plotted in figure 7. It is apparent that the gradation of the material collected from the second extraction more nearly approaches the desired gradation, but the difference between the two curves is very small.

COMPARISON OF LAYER METHOD AND DECANTATION METHOD

Inasmuch as the layer method is considered more similar to the decantation method than to any other, a comparison of these two methods is made for further evaluation of the former method. As have been discussed, the criteria for evaluation are whether the extracted sample is typical, and the amount of time and effort needed to obtain a desired quantity of the sample.

The representativeness of the material obtained from a single extraction by each method is illustrated by schematic diagrams (figure 2). In discussing the layer method of extraction, it was pointed out that the ratio of particle sizes 1, 2, and 3 is 1:1:1, both in the desired fraction and in the material obtained by the layer method. In the decantation method the size ratio is 3:2:1 in the material obtained by a single extraction (figure 2b). The material thus obtained is, therefore, not representative of the fraction desired.

A numerical example may be used to compare further the samples obtained from a single extraction by the two methods. In this example assumptions such as the gradation of the hypothetical soil, the height of the soil suspension, and the size range of the clay material to be separated are the same as in the previous examples.

Both the position and thickness of the layer of soil suspension extracted by the layer method can be varied. For comparison, the layer of case III (table II) will be assumed. The use of the equations in Appendix I for determining the size composition of the material in a layer is shown in Appendix II. In the decantation method of extraction, the soil suspension above Level A-A (figure 1) is removed. For the assumed conditions, level A-A is 8.00 cm. below the surface of the suspension (see section on mathematical analysis). The size composition of the extracted material can also be determined by use of the equations in Appendix I.

TABLE III. COMPARISON OF CLAY-SIZE MATERIAL EXTRACTED BY LAYER AND DECANTATION METHODS

Particle Size, mm. Finer Than	Particle Size, mm. Coarser Than	Percent by Weight in the Desired Clay Fraction	Material Obtained by Layer Method* Weight, g.	Material Obtained by Layer Method* Percent	Material Obtained by Decantation Method Weight, g.	Material Obtained by Decantation Method Percent
0.00225	0.00200		0.12	1.0	0	0
0.00200	0.00175	4.5	0.39	3.4	0.12	0.6
0.00175	0.00150	5.1	0.58	5.1	0.40	2.1
0.00150	0.00100	13.5	1.57	13.8	1.90	10.0
0.00100	0.00060	17.1	1.93	16.9	3.28	17.3
0.00060	0.00030	23.1	2.63	23.0	5.03	26.4
0.00030	0.00010	36.7	4.20	36.8	8.29	43.6
Totals		100.0	11.42	100.0	19.02	100.0

*In both the decantation method and the layer method, the data refer to the material obtained from a single extraction.

The size composition of the materials obtained by the two methods of extraction shown in table III and in figure 8 indicate that a nearly representative sample can be obtained from a single extraction by the layer method. But, since the gradation of the material obtained from a single extraction by the decantation method differs considerably from the desired gradation, repeated withdrawals are necessary to obtain a representative sample by the decantation method.

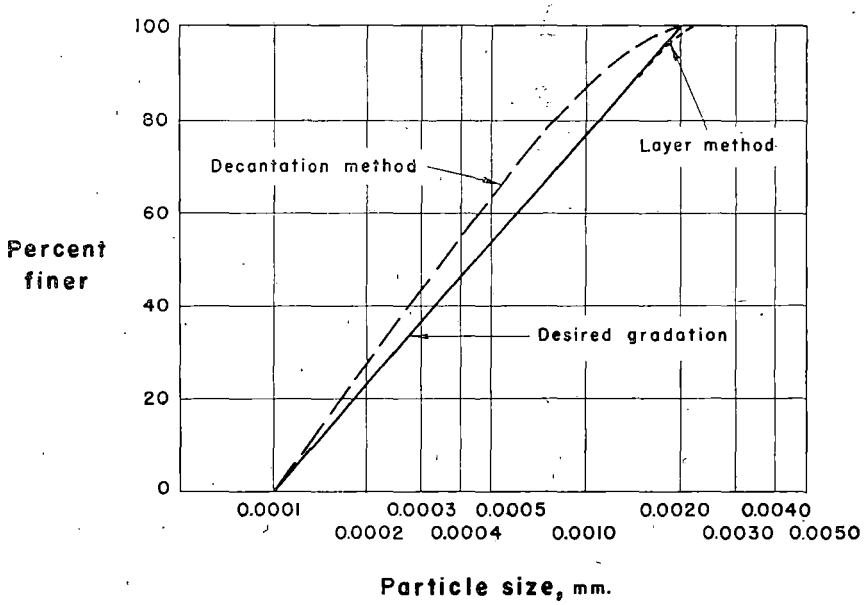

Fig. 8. Particle-size accumulation curves showing difference in gradation of clay-size material obtained from single extractions by the layer method and by the decantation method.

The large number of repeated withdrawals and the amount of water mixed with the clay-size material owing to repeated dilutions make extraction by the decantation method a time-consuming process. The layer method simplifies the process by eliminating the necessity for repeated withdrawals and continuous dilutions without sacrificing the representativeness of the sample obtained. The possible magnification of experimental errors due to the large number of repeated withdrawals may also be avoided in the layer method of extraction.

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APPENDIX I

DERIVATION OF EQUATIONS

The equations presented in this appendix may be used to determine the size composition of materials obtained by the layer and the decantation methods of extraction. The equations are derived on the assumption of gravity sedimentation, although the sedimentation of suspended soil particles can be either by gravity or by centrifugation.

In the layer and the decantation methods of extraction, the procedure involves the removal of a portion of soil suspension from a container. Different kinds of apparatus may be used for this purpose, but any method used causes some disturbance in the desired portion and the suspension adjacent to it. The material obtained by extraction therefore may differ from that actually contained in the desired portion prior to extraction. In the mathematical analysis presented herein, such differences are assumed to be negligible.

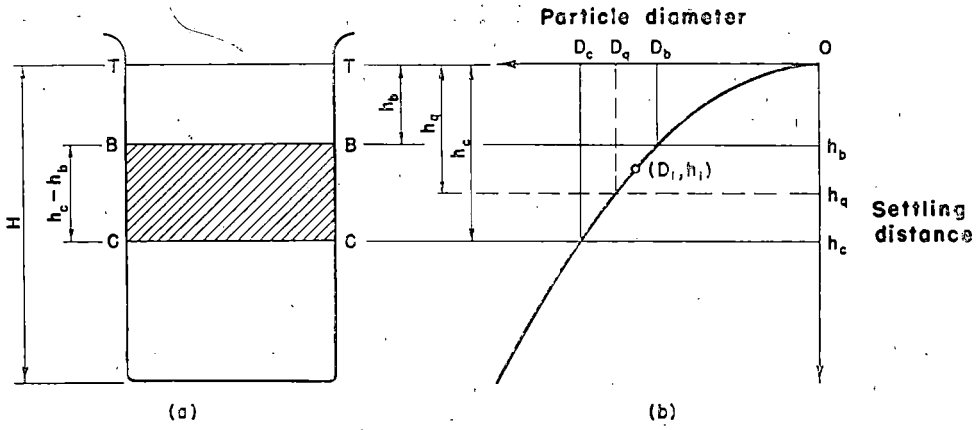

Fig. 9. (a) Shows position and thickness of a layer to be extracted.

Assume that a soil sample is properly dispersed and allowed to settle by gravity in a container (figure 9a). Before sedimentation, particles of all sizes are uniformly distributed throughout the suspending medium. After a given sedimentation period, particles of different sizes will have settled through different distances, which can be determined by Stokes' law. For a given sample, a given sedimentation period, and a given temperature during sedimentation, Stokes' law as expressed by equation (1) can be written as

$$h = KD^2 \tag{3}$$

where h = distance through which soil particles settle.
D = particle diameter.
K is a constant.

Assume that particles of different sizes are at the surface (level T-T) of the soil suspension before sedimentation. Then the position of each of these particles after a given sedimentation period can be determined by computing its settling distance according to equation (3) (figure 9b).

A layer of soil suspension (between levels B-B and C-C) will be extracted from the container after a given sedimentation period (figure 9a). Note that no particles with diameter greater than D_b remain in suspension above level B-B and none with diameter greater than D_c remain in suspension above level C-C at the instant of extraction. It has been mentioned that before sedimentation all particles are uniformly distributed throughout the suspending medium. The amount of soil obtained from the layer at the end of a given sedimentation period therefore will be smaller than the amount of soil suspended in the same layer before sedimentation.

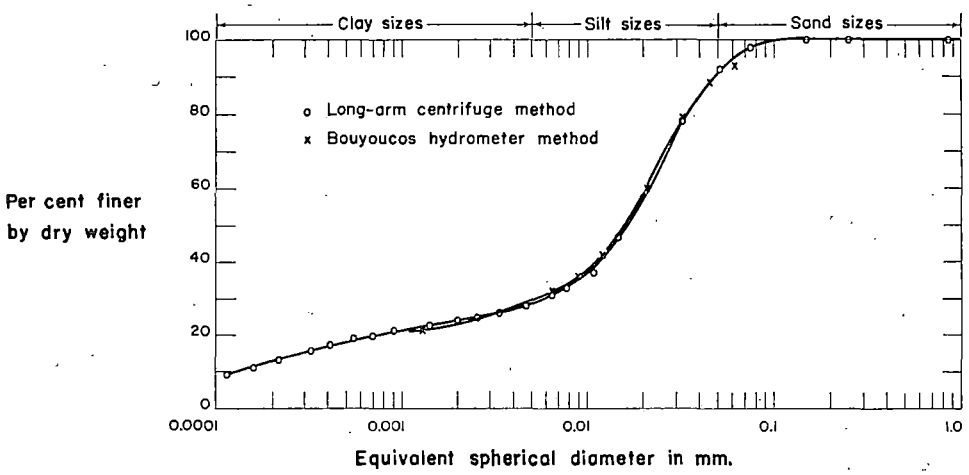

Fig. 9. (b) Shows a curve representing positions of soil particles after a given sedimentation period. These particles were at level *T-T* before sedimentation.

The particle-size accumulation curve is conventionally plotted on semi-logarithmic paper with the particle diameter on the logarithmic scale. This accumulation curve of any soil can be approximated by several straight line segments. The general equation of these segments is

$$P = m \log D + R_1 \qquad (4)$$

where P = percentage of particles finer than D.
D = particle diameter.
m and K_1 are constants.

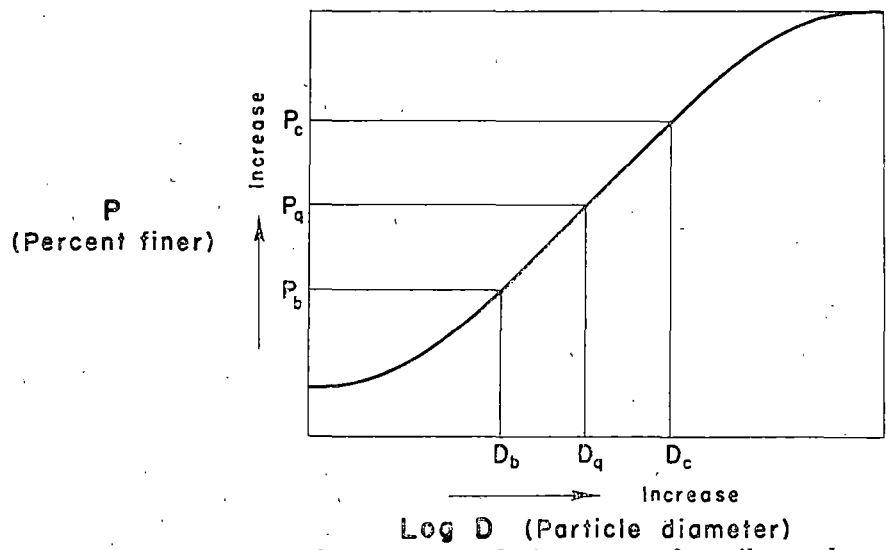

Fig. 10. Particle-size accumulation curve of a soil sample.

Assuming the particle size accumulation curve of a soil to be as shown in figure 10, the section of the curve representing particles with diameters in the range D_b-D_c approaches a straight line and can be represented by equation (4).

Before Sedimentation

Since particles of all sizes are uniformly distributed throughout the suspending medium before sedimentation, the weight of soil particles contained in the layer between level B-B and level C-C can be computed by the following equation:

$$w = \frac{h_c - h_b}{H} \overline{W} \tag{5}$$

where w = weight of soil contained in the layer (including particles of all sizes).

$\overline{W}$ = total weight of dispersed soil.

$h_c - h_b$ = thickness of the layer.

H = total depth of the soil suspension in the container.

If particles with diameter smaller than D_b are considered, their weight can be determined from the equation,

$$w_{fb} = \frac{w P_b}{100} \tag{6}$$

where w_{fb} = weight of particles having diameter smaller than D_b contained in the layer before sedimentation.

P_b = percentage of particles having diameter smaller than D_b in the dispersed sample (figure 10).

If particles in the size range D_b-D_q (figure 10) are considered, their weight can be determined from the equation,

$$w_{bq} = \frac{w}{100} (P_q - P_b) \tag{7}$$

where w_{bq} = weight of particles in the size range D_b-D_q contained in the layer before sedimentation.

P_q = percentage of particles finer than size D_q in the dispersed sample.

Substituting equation (4) in equation (7) and simplifying,

$$w_{bq} = \frac{w\, m}{100} \log \frac{D_q}{D_b} \tag{8}$$

Equations (7) and (8) are valid when D_q is within the range D_b-D_c because only the segment of the particle-size accumulation curve representing this size range approaches a straight line.

After a Given Sedimentation Period

Figure 9b shows that those particles with diameter smaller than D_b which were at level T-T before sedimentation have settled through certain distances after a given sedimentation period, but their new positions are still above level B-B. Particles finer than size D_b are therefore still uniformly distributed throughout the layer at the instant of extraction, and equation (6) can be used to compute the weight of particles obtained from the layer.

The weight of particles in the size range D_b-D_q (figure 9) suspended in the layer will be less than that computed by equation (7) because particles in this size range are no longer uniformly distributed throughout the layer at the end of the sedimentation period. The settling distance of particles of size D_i is h_i (figure 9). Therefore, at the end of the sedimentation period no particles of size D_i remain in suspension above a level which is h_i below level T-T. Particles of size D_i in the layer between levels B-B and C-C remain in suspension only in the lower portion. The thickness of this lower portion is (h_c-h_i), while the thickness of the layer is (h_c-h_b).

The weight of soil particles of an infinitesimal size range embracing size D_i which remain in suspension in the layer between levels B-B and C-C at the end of the given sedimentation period will be

$$\frac{h_c-h_i}{h_c-h_b} \times \frac{w \Delta P_i}{100}$$

where P_i is the percentage by weight of the particles within the infinitesimal size range in the dispersed sample. By a process of summation, the following expression can be derived:

$$w_{bq}' = \lim_{m \to \infty} \sum_{1}^{M} i = \frac{h_c-h_i}{h_c-h_b} \frac{w \Delta P_i}{100}$$

$$\text{or} \quad w_{bq}' = \frac{1}{100} \int_{P_b}^{P_q} \frac{h_c-h}{h_c-h_b} w \, dP \qquad (9)$$

where w_{bq}' = weight of particles in the size range D_b-D_q contained in the layer at the end of the sedimentation period. In this analysis, w_{bq}' is also the weight of particles in the size range D_b-D_q obtained from the layer.

From equation (4),

$$dP = \frac{m \log e}{D} d D \qquad (10)$$

Substituting equations (3) and (10) in equation (9),

$$w_{bq}' = \frac{w\ m \log e}{100\ (h_c - h_b)} \int_{D_b}^{D_q} \frac{(h_c - KD^2)}{D}\ d\ D$$

Integrating

$$w_{bq}' = \frac{w\ m \log e}{100\ (h_c - h_b)} \left[h_c \ln \frac{D_q}{D_b} - \frac{K\ (D_q^2 - D_b^2)}{2} \right]$$

Substituting $h = K D^2$ $\log D = \log e \ln D$ and simplifying

$$w_{bq}' = \frac{w\ m}{100\ (h_c - h_b)} \left[h_c \log \frac{D_q}{D_b} - \frac{(h_q - h_b) \log e}{2} \right] \quad (11)$$

From equations (8) and (11)

$$\frac{w_{bq}'}{w_{bq}} = \frac{1}{h_c - h_b} \left[h_c - \frac{(h_q - h_b) \log e}{2 \log \frac{D_q}{D_b}} \right] \quad (12)$$

Equation (12) gives the ratio of the weight of particles in the size range D_b-D_q obtained from the layer after a given sedimentation period to the weight of particles in the same range prior to sedimentation.

It will be noted that equations (11) and (12) are valid only when D_q is within the size range D_b-D_c and the segment of the particle-size accumulation curve representing this size range approaches a straight line. If the segment of the curve differs significantly from a straight line, a similar approach for equation derivation can be followed except that the segment of the curve should be either approximated by a number of straight line segments or represented by a non-linear equation.

APPENDIX II

EXAMPLE OF CALCULATIONS

The methods of calculation of the size composition of clay-size material obtained by the layer and the decantation methods of extraction are practically the same. In the example which follows only the computation relating to the layer method is presented.

The gradation of a hypothetical soil has been shown in table I and figure 3. The soil is dispersed in distilled water, and after a sedimentation period of six hours a layer positioned as in case I (table II) is removed from the soil suspension. The size composition of the material obtained from the removed layer is to be determined.

Data relating to the soil suspension have been discussed under the heading "Mathematical Analysis." The pertinent values are summarized as follows:

$\overline{W} = 150$ g.
$H = 21.00$ cm.
$h_b = 4.50$ cm.
$h_c = 8.00$ cm.
$D_b = 0.0015$ mm.
$D_c = 0.002$ mm.
$P_b = 36.2$
$P_c = 40.0$

The significance of each symbol is given in Appendix I.

Before computing the weight of particles in any specific size range, constants m and K_1 must be determined. [See equation (4).] From the gradation of the hypothetical soil given in table I,

$P = 0$ for $D = 0.00010$ mm.
$P = 30.7$ for $D = 0.00100$ mm.

Substituting in equation (4) and solving simultaneously for the two unknowns m and K_1, we obtain

$m = 30.7$
$K_1 = 122.8$

We then have for equation (4)

$$P = 30.7 \log D + 122.8 \tag{13}$$

In determining the size composition of clay-size material obtained from the layer, several control sizes should be selected. By computing the weight of material finer than each control size, the particle-size accumulation curve of the extracted material can be plotted. The control sizes used in this example are 0.002 mm., 0.00175 mm., 0.0015 mm., 0.001 mm., 0.0006 mm., 0.0003 mm., and 0.0001 mm.

The data and figure 9b show that the material finer than 0.00150 mm. is still uniformly distributed throughout the layer at the instant of extraction. The material in this size range obtained from the layer can therefore be computed by combining equations (5) and (6) to obtain

$$w_{fb} = \frac{h_c - h_b}{100\,H}\,\overline{W}\,P_b$$

Substituting $h_c = 8.00$ cm.
$h_b = 4.50$ cm.
$H = 21.00$ cm.
$\overline{W} = 150$ g.
$P_b = 36.2$
we get $w_{fb} = 9.05$ g.

The gradation of this material is the same as that of the fraction finer than 0.0015 mm. in the hypothetical soil. The weight of material finer than 0.001 mm. obtained from the layer can then be computed as follows:

$$\text{Weight of minus 0.001 mm. material} = 9.05 \times \frac{30.7}{36.2} = 7.68 \text{ g.}$$

Where 30.7 and 36.2 are the percentages of material finer than 0.00100 mm. and 0.00150 mm. respectively in the hypothetical soil (table I).

Similarly, the weights of minus 0.00060 mm. and minus 0.00030 mm. materials obtained from the layer can be computed and results are given in table IV. The weight of minus 0.00010 mm. material obtained from the layer will be zero because the hypothetical soil contains no particles finer than 0.00010 mm.

Since $D_c = 0.002$ mm., figure 9b shows that all the material obtained from the layer will be finer than 0.002 mm. The weight of material in the size range 0.002 - 0.0015 mm. obtained from the layer can be computed by combining equations (5) and (11) to obtain

$$w_{bq}' = \frac{m\overline{W}}{100\,H}\left[\, h_c \log \frac{D_q}{D_b} - \frac{(h_q - h_b)\log e}{2} \,\right] \qquad (14)$$

Substituting $m = 30.7$ as given in equation (13).
$\overline{W} = 150$ g.
$H = 21.00$ cm.
$h_q = h_c = 8.00$ cm.
$D_q = D_c = 0.002$ mm.
$h_b = 4.50$ cm.
$D_b = 0.00150$ mm.
we get $w_{bq}' = 0.53$ g.

Hence

Weight of minus 0.002 mm. material
$= w_{fb} + w_{bq}' = 9.05 + 0.53 = 9.58$ g.

In a similar manner, the weight of material in the size range 0.00175 - 0.00150 mm. obtained from the layer can be computed by using equation (14). In this example,

$$D_q = 0.00175 \text{ mm.}$$

From equation (2),

$$= 2.00 \times 10^6 D_q^2$$
$$= 2.00 \times 10^6 (0.00175)^2 = 6.12 \text{ cm.}$$

The values of m, $\overline{W}$, H, h_c, D_c, h_b, and D_b are the same as before. Substituting to equation (14), the weight of material in the size range 0.00175 - 0.0015 mm. is found to be 0.40 g.

Weight of minus 0.00175 mm. material $= 9.05 + 0.40 = 9.45$ g. From

Appendix I it will be noted that the weight of the material in the size ranges 0.002 - 0.0015 mm. and 0.00175 - 0.0015 mm. can also be computed by combining equations (7) and (12).

TABLE IV. SIZE COMPOSITION OF CLAY-SIZE MATERIAL OBTAINED FROM CASE I LAYER

Particle Size, mm.	Weight of material finer than size shown, g.	Percent of material finer than size shown
0.00200	9.58	100
0.00175	9.45	98.7
0.00150	9.05	94.5
0.00100	7.68	80.2
0.00060	5.98	62.5
0.00030	3.68	38.4
0.00010	0	0

The weights of material finer than each control size are summarized in table IV. To plot the particle-size accumulation curve for the material obtained from the layer, the size composition of the material is expressed on a percentage basis in the same table (figure 4).

PIPETTE METHOD
TO SUPPLEMENT HYDROMETER TESTS
FOR PARTICLE-SIZE DETERMINATION IN SOILS

by

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(Highway Research Board Proceedings, 32:548-555. 1953.)

PIPETTE METHOD

In investigations carried on with loess soils, a comparatively simple test, a pipette method with gravity settling, was developed to supplement the standard hydrometer test for determining particle size distribution in the clay range below 0.005 mm. and for extending measurements below 0.001.

The behavior characteristics of soils are governed to a large extent by the clay minerals present. Since particle size distribution below 0.005 mm. is somewhat diagnostic of the kinds of clay minerals found in soils, measurements in this size range are important in the study of the clay fractions of soils.

To measure particle sizes in the silt and clay ranges down to 0.001 mm., the hydrometer test is used in civil engineering soil laboratories because of its simplicity and rapidity. For such measurements in agricultural soil laboratories, the more complex and time consuming pipette method[14] has been adopted. Bouyoucos[2, 3, 4, 5, 6] has reported close agreement between the results of the hydrometer and the pipette tests.

Particle size determinations in the clay range finer than 0.001 mm. have been somewhat neglected due to the difficulty of measurement. The standard hydrometer test and the usual pipette methods employing gravity settling are unsatisfactory because of the long settling time necessary. A centrifuge is generally used to speed up settling[16], and a centrifuge hydrometer method has been developed[9, 13]. In these methods the effects of swirling and currents in the centrifuge tube are questioned by some authorities. A special centrifuge with a sector shaped tube was developed to achieve radial settling without interference from the straight-walls of the ordinary centrifuge tube[10]. A two layer method involving two liquids in a centrifuge tube has also been developed which, while shown to be accurate, is rather time consuming[11, 12].

A comparatively simple test was desired to supplement the standard hydrometer test for determining the particle size distribution in the clay

range below 0.005 mm. and for extending measurements below 0.001 mm. A pipette method with gravity settling was developed for this purpose.

Basic Principles of Settlement Analysis

The settling velocity of spheres of equal size, falling through a medium, may be calculated from Stokes' law; and from the velocity, the distance and time of settling may be determined. In a soil suspension, the time is measured from when the suspension was last stirred, and the depth or distance of settling is measured from the surface of the suspension. The calculated diameter of particles falling through this distance is then the maximum diameter of particles remaining at that depth. Since smaller particles are still in suspension at that depth, their concentration is a measure of the percent of particles smaller than the specified diameter. The hydrometer and pipette are used to measure this concentration of particles.

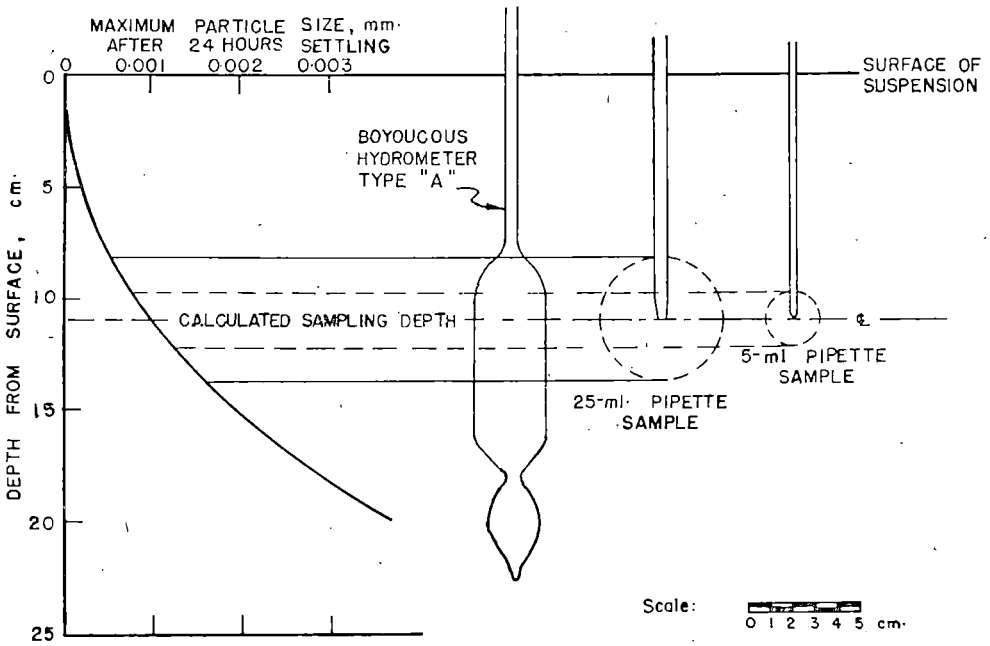

Fig. 1. Graph shows particle size at left in depth ranges covered by hypothetical hydrometer and pipette measurements after 24 hr. of settling.

Hydrometer

Particle concentrations are determined with the hydrometer by measuring the specific gravity of the suspension (figure 1). The hydrometer does not measure the specific gravity at any particular depth but through a

range of depths in the suspension. Concentrations of larger and smaller particles lower and higher in the suspension will thus affect the reading, and the particle concentrations measured will be over a range of particle sizes instead of being just one size. However since particle size accumulation curves in natural soils are usually smooth, this is not too serious an error. Hydrometers of the type illustrated (figure 1) have been calibrated on the basis of pipette tests, and there is fairly close agreement in results obtained with both[18].

Pipette

In the pipette test instead of measuring the specific gravity of the suspension, a sample of known volume is withdrawn at the specified depth; then it is dried and weighed. Although much criticism has been directed at the hydrometer test because it measures particle size ranges, not particle sizes, it is usually overlooked that the pipette does this also. A sphere of material is withdrawn from the suspension, but the smaller the pipette the smaller the range of depths samples.

Measurement of Colloidal Sizes

A number of authors have pointed out that gravity settling of a suspension is not practical for particle size determinations below 0.001 or possibly 0.0005 mm. due to the effect of Brownian movement and convection currents arising from slight changes in temperature. However, gravity sedimentation has been used for determinations down to 0.0005 mm. extended the lower limit to 0.0001 mm. in a study of colloidal suspensions of gold[16, 17].

Colloidal particles in a suspension will reach equilibrium when the force of gravity on the particles is counterbalanced by the kinetic forces resulting from greater particle concentrations lower in the suspension. After equilibrium is reached there will be far fewer particles in suspension near the surface than deeper in the suspension. Therefore a shallow sampling depth will tend to minimize the effects of colloidal interference in the settling. In addition to the colloidal effect, other errors are present in unknown amounts. These errors include the assumption of a spherical particle shape in the application of Stokes' law, and the uncertain effective specific gravity of the clay particles, which may vary with the amount of adsorbed water and the kind of clay mineral.

SUPPLEMENTAL PIPETTE METHOD

Apparatus

With the pipette apparatus, the lower limit of the clay size range was extended to 0.0002 mm. A 5 ml. automatic pipette fastened in a Shaw pipette rack was used. The rack was swivelled to a ring stand in such a way that sedimentation cylinders could be set in a circle around the stand. This

arrangement permits the settling of up to 12 suspensions at one time. The soils to be tested were dispersed according to the ASTM standard method (ASTM Designation: D422-39) as modified[8], using sodium metaphosphate as the dispersing agent. Standard 1000 ml. glass graduates are used, and the air jet dispersion apparatus is well adapted to the method[7].

Settling Time and Sampling Depth

The time of settling for spherical particles of any diameter may be calculated from Stokes' law, expressed as

$$T = \frac{30\eta L}{980\, d^2 (G - G_1)},$$

where T is the time in minutes, η the viscosity of the settling medium in poises, L the depth in centimeters, d the particle diameter in millimeters, and G and G_1 the specific gravities of the particles and the settling medium respectively.

The specific gravity of the soil particles, G, should represent material in the size range being studied. Since only the specific gravity of the minus 10 sieve portion of the soil is usually determined in engineering laboratories, this value is used in the ASTM standard hydrometer test. It may be used in the supplemental pipette test, and the error will probably be in keeping with the limitations of Stokes' law. In working with the clay fraction, the error will vary with the types of clay minerals present.

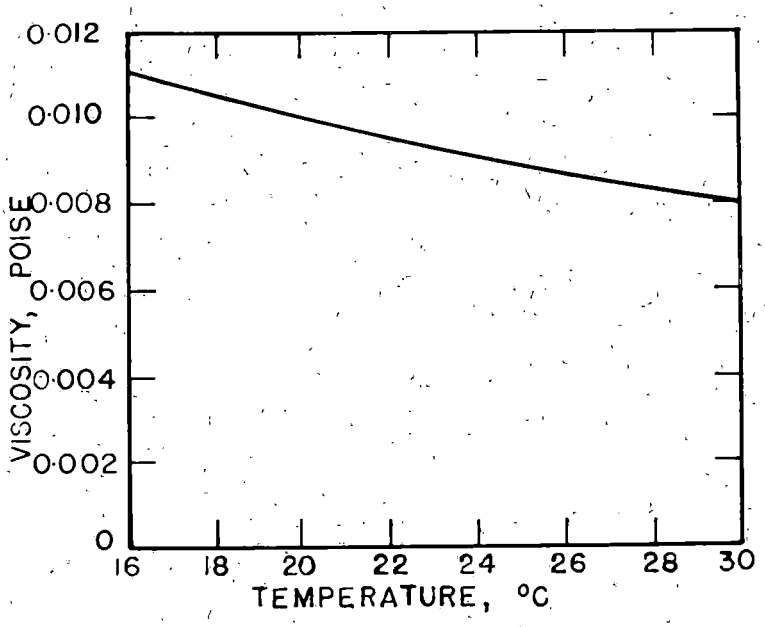

Fig. 2. Relation of viscosity of water to temperature.

Since η, the viscosity of water, varies with temperature (figure 2), adequate temperature control of the sedimentation cylinders is necessary. The control is either by immersing in a water bath or by keeping the cylinders in a room with a fairly constant temperature. Variations in temperature

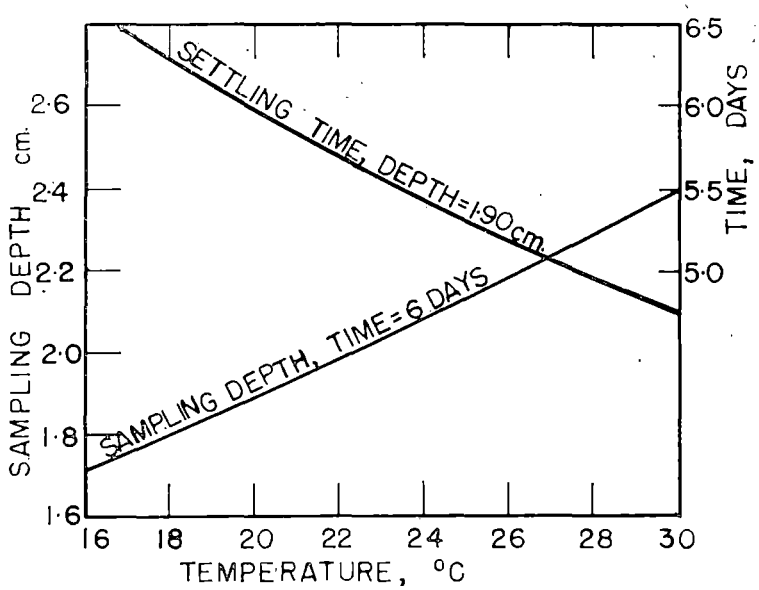

Fig. 3. Settling times with a constant depth or sampling depth with a constant time as influenced by variations in temperature: d equals 0.002 mm., G equals 2.70.

during the settling period may be compensated for by adjustments of the settling time or of the sampling depth (figure 3). If the variation is not great, the average temperature over the settling period may be used in calculations. Ordinarily samples are taken at predetermined depths, and the time is varied to compensate for changes in temperature. With adequate temperature control this is not necessary. Following is an example of time and depth calculations from Stokes' law: If

$$T = \frac{30\eta L}{980\, d^2 (G - G_1)},$$

the average temperature throughout settling is 67° F. (19.4° C), η=0.0102 poise. If d=0.002 mm., L=5 cm., G= 2.60,

$$T = \frac{30(0.0102)\ (5)}{980(0.002)^2\ (2.60 - 1.00)} = 244 \text{ min.} = 4 \text{ hr. } 4 \text{ min.}$$

Sampling

Sampling with the pipette may be done at shallow depths to lessen the settling time. However, since a sphere of material is withdrawn from the

suspension, the sampling depths must be at least equal to the radius of the sphere. A small volume pipette will thus allow shallower sampling depths. Since weighing errors will increase with decreasing sample size, a 5 ml. pipette is probably the minimum practical size (table I).

TABLE I. A SAMPLING SCHEDULE FOR PIPETTE ANALYSIS

Equivalent spherical particle diameter	Average temperature	Sampling depth	Settling time
mm.	C.	cm.	
0.002	20	5.0	3 hr. 46 min.
0.001	20	3.0	9 hr. 5 min.
0.0005	20	2.0	24 hr. 10 min.
0.0002	20	1.9	6 days

The Automatic Pipette

Before using the pipette it is calibrated with mercury to determine its exact capacity. The pipette is filled to the level of the stopcock. Turning the stopcock 180 degrees allows the material to flow out.

In the pipette method, as previously discussed, samples are taken at predetermined depths. To obtain a uniform soil water mixture at the start of the settling period the suspension is shaken by hand in 1000 ml. glass graduates, according to the ASTM standard method[1].

The Shaw pipette rack provides an accurate method of lowering the pipette a set distance into the suspension. When the pipette is at the desired sampling depth, suction is applied by means of an aspirator bottle adjusted so that the filling time is about 8 seconds. Because successively shallower sampling depths are used, reshaking the suspension between samplings was unnecessary. Considerable time is saved by this procedure[15, 16].

After the pipette is filled and withdrawn from the soil suspension, it is emptied into a 12 or 15 ml. weighing bottle. Particles clinging to the inside of the pipette are washed into the bottle with distilled water. The pipette is rinsed with acetone to speed drying and is connected to an aspirator or vacuum line to dry it. Weighing bottles must be washed, oven dried and weighed again before reusing.

The filled weighing bottle, after being dried in a constant temperature (105° to 110° C.) oven, is weighed on an analytical balance. The oven dry weight of the sample must be corrected for the amount of dispersing agent present. Since sodium metaphosphate remains hydrated at oven dryness, the correction may be obtained by oven drying a pipetteful of sodium metaphosphate solution of the same concentration used in the soil suspension. The corrected weight of the sample can then be obtained and used to calculate the percentage by weight of particles finer than the maximum particle size measured. The calculation procedure is illustrated by the following example:

Total wt. of air-dry soil used in test	50.0 gm.
Hygroscopic moisture content	5.00%
Wt. of soil in an oven-dry condition	47.6 gm.
The pipette sample was taken to measure material finer than 0.002 mm.:	
Wt. of oven-dried pipette sample and bottle	19.1644 gm.
Oven-dry wt. of bottle	19.0722 gm.
Wt. of oven-dry pipette sample	0.0922 gm.
Calibrated volume of the pipette	5.025 ml.
Oven-dry wt. of dispersing agent in 5.025 ml. of solution	0.0159 gm.
Wt. of oven-dry soil in pipette sample	0.0922 − 0.0159 = 0.0763 gm.

Oven-dry wt. of soil particles finer than 0.002 mm.

in the whole suspension $(0.0763)\frac{1000}{5.025} = 15.19$ gm.

Percent by weight of the soil particles finer than 0.002 mm $(100)\frac{15.19}{47.6} = 31.8\%$

Results of Combined Pipette Hydrometer Analysis

Particle size accumulation curves for two loess soils with high and low clay contents show the smooth transition between hydrometer and pipette test data (figure 4). The data in the range where the test methods overlap show close agreement of hydrometer and pipette test values for a number

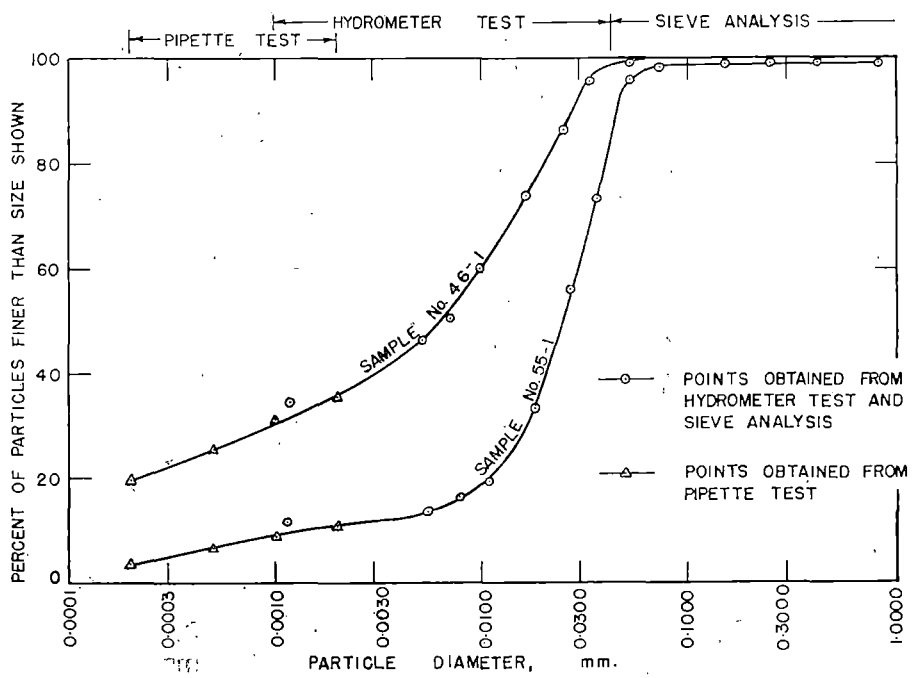

Fig. 4. Particle size accumulation curves for soils tested which had the lowest (No. 55-1) and highest (No. 46-1) clay contents.

TABLE II. CLAY CONTENTS BY HYDROMETER AND PIPETTE TESTS*

Sample No.	Clay Contents from Hydrometer Test, % oven-dry wt.†		Clay Contents from Pipette Test, % oven-dry wt.			
	0.002 mm.	0.001 mm.	0.002 mm.	0.001 mm.	0.0005 mm.	0.0002 mm.
55-1	12.0	11.4	10.5	8.6	6.5	3.8
20-2	16.0	14.5	15.4	12.7	9.3	4.0
61-2	19.6	18.0	18.8	15.7	11.2	6.9
26-1	22.4	20.0	21.5	18.1	14.2	8.3
26-2	23.1	19.8	23.4	19.8	15.1	7.8
29-1	25.0	22.2	24.0	20.7	16.6	11.4
36-1	28.9	25.0	28.4	24.6	20.5	14.3
43½-1	33.0	29.8	31.4	27.6	23.2	17.3
46-1	36.2	32.0	35.3	31.2	25.4	19.8
49-3	31.4	28.4	28.2	25.6	20.5	12.8
49-4	24.6	22.4	22.8	19.7	15.8	10.5

*All clay contents are in terms of percent finer than particle diameter indicated.
†All hydrometer test values are interpolated from particle size accumulation curves.

of loess soils (table II). Corresponding values taken from the hydrometer tests and from the pipette tests for the 0.002 mm. clay vary up to 3.2 percent, the average difference in values being 1.2 percent. In the 0.001 mm. clay size, the average difference is 1.7 percent. For both the 0.001 mm. and 0.002 mm. sizes the hydrometer test values tend to be higher. (All pipette tests were run in duplicate, and the results checked within 1 percent. In normal testing procedure duplicate tests would not be necessary.)

As previously mentioned, the shapes of particle size accumulation curves in the clay range should be an indication of the types of clay minerals present. The particle size curves in figure 5 illustrate the effect the kind of clay

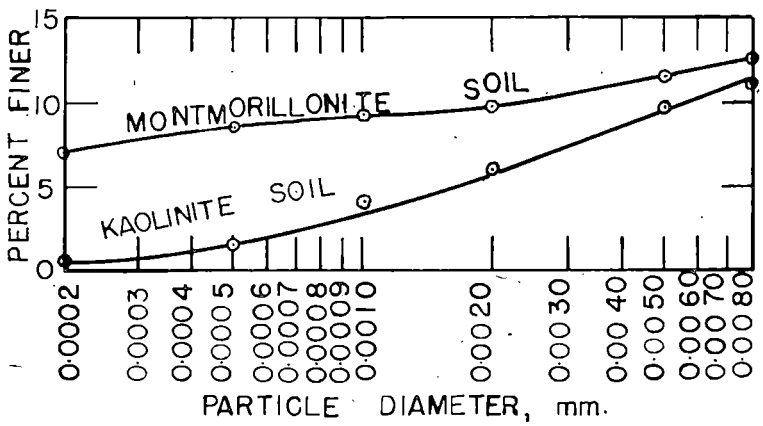

Fig. 5. Particle size accumulation curves for the clay range of synthetic montmorillonite and kaolinite soils.

mineral has on the particle size distribution of two synthetic soils so made as to have approximately equal amounts of 0.005 mm. clay. The clay minerals in the two soils are kaolinite in one and montmorillonite in the other, and the nonclay portions of both are cleaned quartz. Much more work is needed with relatively pure clay minerals and known mixtures of clay minerals before the significance of the shape of the particle size curve in the clay range of soils can be fully interpreted. This approach to clay mineral identification should prove to be a useful supplement to other identification methods.

With two exceptions (samples 49-3 and 26-2) the shapes of the particle size accumulation curves for the clay range of some of the loess soils tested by the combined pipette hydrometer method are markedly similar, though they represent soils having 0.005 mm. clay contents varying from 13 to 45 percent (figure 6). From differential thermal analyses[9], the clay minerals

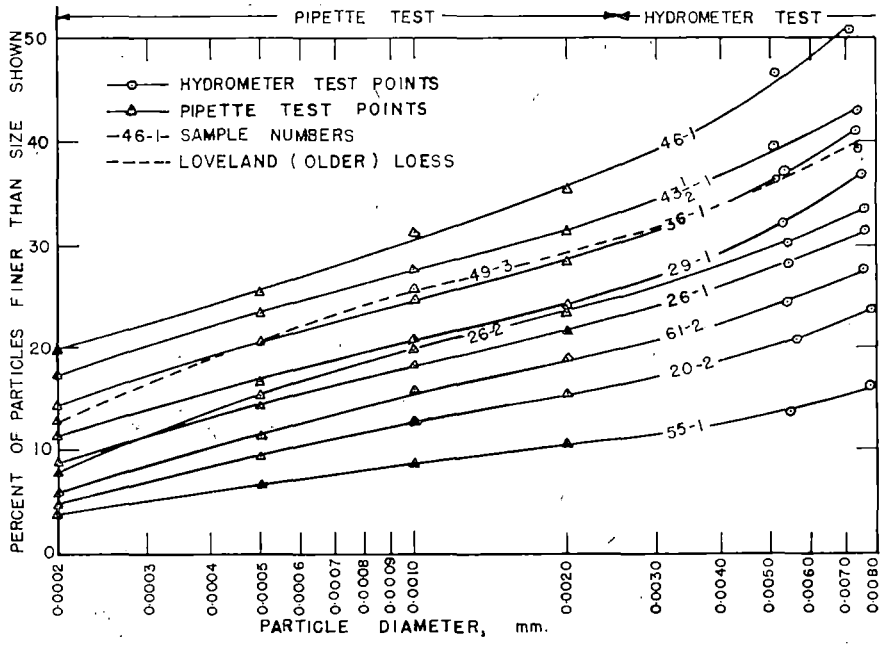

Fig. 6. Particle size accumulation curves for the clay range of some of the loess tested.

in these soils are the same, being probably illite or a mixture of illite and montmorillonite. The shapes of the particle size curves appear to confirm this. Sample 26-2 differs in that it is gray-colored and unoxidized and the other samples are buff colored and oxidized. Sample 49-3 is an older, highly weathered soil. Other data may affect size analysis of these soils (table III).

TABLE III. PROPERTIES OF SOILS TESTED WHICH MAY AFFECT MECHANICAL ANALYSIS

Sample No.	Material	Age	Location (All in SW Iowa) (County)	Sampling Depth	P.I.	Cation Exchange Capacity	Organic Matter	$CaCO_3$ Content	Engineering Classification (AASHO)
				ft.	%	me./100 gm.	%	%	
55-1	Loess	Upper Wisconsin (?)	Harrison	2-3	2.3	11.2	0.24	11.0	A-4 (8)
20-2	Loess	Wisconsin	Harrison	39-40	6-2	13.4	0.17	10.2	A-4 (8)
61-2	Loess	Wisconsin	Ida	17-18	10.8	14.2	0.15	11.5	A-6 (8)
26-1	Loess	Wisconsin	Shelby	4-5	12.5	18.2	0.18	1.4	A-6 (9)
26-2	Loess	Wisconsin	Shelby	10-11	17.8	17.9	0.17	3.6	A-6 (9)
29-1	Loess	Wisconsin	Audubon	5-6	18.0	19.5	0.25	2.9	A-6 (11)
36-1	Loess	Wisconsin	Montgomery	5½-6½	20.8	21.0	0.21	1.8	A-7-6 (13)
43½-1	Loess	Wisconsin	Fremont	4½-5½	33.4	24.4	0.37	0.5	A-7-6 (18)
46-1	Loess	Wisconsin	Page	5-6	32.7	22.6	0.30	1.5	A-7-6 (19)
49-3	Weathered Loess Soil	Loveland	Pottawattamie	55-56	24.6	22.7	0.19	1.2	A-7-6 (15)
49-4	Loess	Loveland	Pottawattamie	65-66	15.3	16.6	0.11	7.0	A-6 (10)

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CALCULATION OF STANDARD PROCTOR DENSITY AND OPTIMUM MOISTURE CONTENT FROM MECHANICAL ANALYSIS, SHRINKAGE FACTORS, AND PLASTICITY INDEX

by

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(Highway Research Board Proceedings, 29:477-481. 1949.)

INTRODUCTION

A new approach to the soil density problem was presented in 1948.* The approach was a mathematical one for determining the standard Proctor density *(maximum dry density in pounds per cubic foot obtained by the standard Proctor laboratory test)* and optimum moisture content. It is based on the premise that the Proctor test gives a density close to the density achieved when a wet soil is allowed to shrink under natural forces to the shrinkage limit. They calculated this density in pounds per cubic foot from the mechanical analysis and shrinkage test data of the soil. The density formula used is

$$\text{Calculated Density} = \frac{D}{1 + \frac{D\text{-}C}{62.5G}} \qquad (1)$$

Where:

$C = 62.5\ R$

$R =$ Shrinkage ratio

$D = C \div \frac{B}{A}$

$A =$ Percent of soil passing U. S. No. 4 sieve

$B =$ Percent of soil passing U. S. No. 40 sieve

$G =$ Approximate specific gravity as calculated from the shrinkage limit and the shrinkage ratio

Formula (1) may be written in more convenient form as

$$\text{Calculated Density} = \frac{6250}{S\left(\frac{B}{A} - 1\right) + \frac{100}{R}} \qquad (2)$$

$S =$ Shrinkage limit

*Rowan, W. H. and Graham, W. W. Proper Compaction Eliminates Curing Period in Constructing Fills. Civil Engineering. 18:450-451. 1948.

The optimum moisture content in percent by dry weight of soil was calculated from the mechanical analysis and shrinkage limit data by means of the formula

$$\text{Calculated Optimum Moisture} = S\,(\frac{B}{A}) \qquad (3)$$

The results of experiments on ten soils indicated that the calculated values agreed fairly closely with those secured by means of the Proctor laboratory tests. The greatest deviation in density between calculated and laboratory results was approximately 5 percent. This was considered within the limits of accuracy, and no correction was deemed necessary. However, the calculated percentage of moisture was usually 1 to 5 per cent higher than the Proctor optimum moisture figure; so an arbitrary correction factor of 3 percent was subtracted from all calculated percentages.

The calculation procedure of Rowan and Graham offered the possibility of eliminating the time and labor involved in the laboratory method, especially for investigations of a routine nature. However, the amount of supporting data did not warrant unqualified acceptance of their procedure. Since only ten soils were tested, all of which were probably indigenous to the Southeastern part of the United States, a more comprehensive verification was considered necessary before full confidence could be placed in such a new development.

Test Data

The verification of the density and optimum moisture formulas was accomplished by comparing the calculated and laboratory values of 210 soils from widespread geographical localities in the United States. The test data from these soils were obtained from three major sources: personal laboratory tests, the files of the Iowa State Highway Commission and the files of the U. S. Bureau of Public Roads. The personal tests were performed on seven Iowa soils and one Virginia soil. Test data on 92 soils representing 28 Iowa counties were furnished by the Iowa State Highway Commission, and the Bureau of Public Roads provided test data on 110 soils sampled in ten different states. It is believed that all data used were obtained by means of the test procedures of the American Association of State Highway Officials.

DEVELOPMENT OF MODIFIED CALCULATION PROCEDURE

Application of the density and optimum moisture formulas to the three different groups of soil test data did not result in the same degree of correlation between calculated and laboratory values as previously found by Rowan

and Graham. As a whole the results were so inconsistent and often so much in error that the validity of the formulas was questioned. When the soil test data were segregated into the groups used in the Public Roads soil classification system, only the calculated densities and optimum moisture contents of the soils in the A-2 and A-4 groups compared favorably with the results of the standard Proctor control tests.

It was further noted that the greatest errors in calculated density were obtained when the formula was applied to the highly plastic soils. To investigate this relationship the percentage error between the calculated and laboratory densities of each soil was determined as follows:

$$\text{Percentage Error} = \frac{\text{Calculated Density} - \text{Laboratory Density}}{\text{Calculated Density}} \times 100$$

These figures were plotted graphically against those for the plasticity index (figure 1).

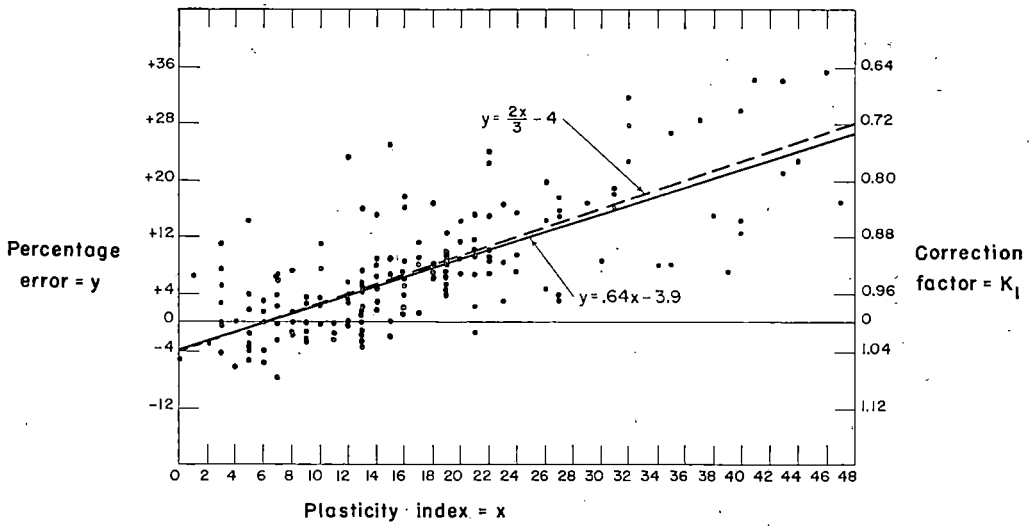

Fig. 1. Correlation between calculated and standard Proctor densities.

The data of the scatter diagram appeared to have a straight line trend. This suggested that a curve might be fitted to the plotted points, and that such a curve could be used to apply a correction factor to the density formula of Rowan and Graham. By such a procedure the calculated density would be brought into closer agreement with the standard Proctor test density.

The curve (solid line in figure 1) was fitted by the least squares method. Its formula is

$$y = .64x - 3.9 \tag{4}$$

where y is the percentage error, and x is the plasticity index. The standard error of estimate was computed to be 6 percent. It is suggested that formula (4) be arbitrarily simplified to read

$$y = \frac{2x}{3} - 4 \tag{5}$$

This simplified form is easier to use and is believed to be in keeping with the accuracy of the data of the scatter diagram from which it was derived. The curve representing formula (5), shown by the dashed line in figure 1, very nearly coincides with the curve of formula (4) through the plasticity index range 0 to 16.

The percentage error can be used to determine the density correction factor K_1 by means of the formula

$$K_1 = \frac{100 - y}{100} \tag{6}$$

which, when expressed in terms of the plasticity index, becomes

$$K_1 = \frac{312 - 2x}{300} \tag{7}$$

The value of K_1 may also be taken directly from the dashed line curve in figure 1.

The modified density formula is obtained by multiplying formula (2) by the correction factor K_1. It may be written

$$\text{Calculated Density} = \frac{6250\ K_1}{S\left(\frac{B}{A} - 1\right) + \frac{100}{R}} \tag{8}$$

An approach similar to that described above was employed to achieve closer agreement between the calculated and laboratory values of optimum moisture content. As in the case of the density computations, the greatest deviations from the Proctor test values occurred with the highly plastic soils. To establish a trend for this error, the deviation between the calculated and laboratory optimum moisture contents of each soil was determined as follows:

$$\text{Deviation (Optimum Moisture)} = \text{Calculated} - \text{Laboratory}$$

The figures were plotted as a scatter diagram graph against those for plasticity index (figure 2).

The data of this scatter diagram showed a linear relationship also, and

a curve (solid line, figure 2) was again fitted by the least squares method. The formula for this curve is

$$z = -.33x + 3.9 \tag{9}$$

where z is the deviation in optimum moisture content, and x is the plasticity

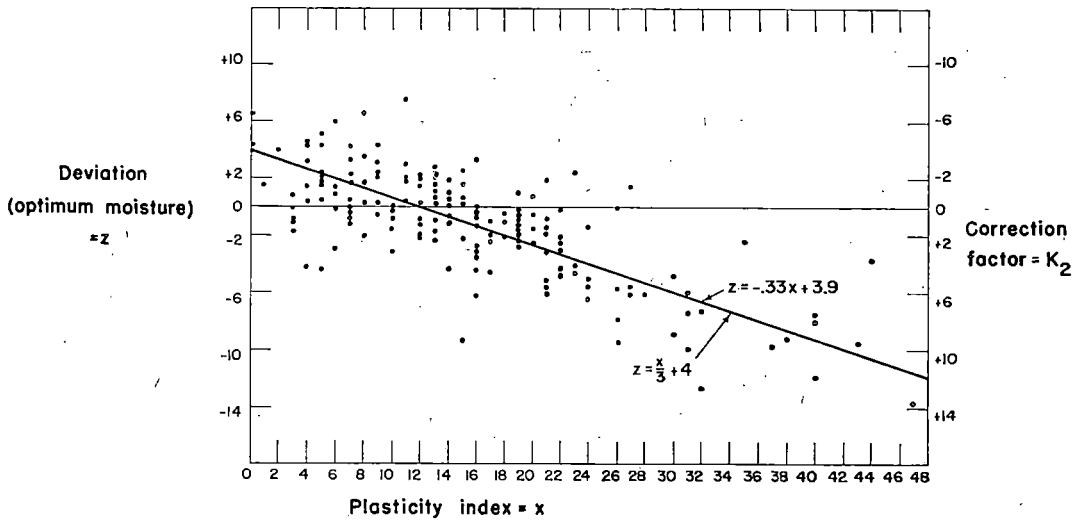

Fig. 2. Correlation between calculated and standard Proctor optimum moisture contents.

index. The standard error of estimate was computed to be 2.5 percent. Formula (9) may be arbitrarily simplified to read

$$z = -\frac{x}{3} + 4 \tag{10}$$

The curve representing this formula practically coincides with the curve of formula (9).

The deviation in optimum moisture content can be used to determine the optimum moisture correction factor K_2 by means of the formula

$$K_2 = -z \tag{11}$$

which, when expressed in terms of the plasticity index, becomes

$$K_2 = \frac{x}{3} - 4 \tag{12}$$

If desired the value of K_2 may be taken directly from the curve in figure 2.

The modified optimum moisture formula is obtained by adding the correction factor K_2 to formula (3). It may be written

$$\text{Calculated Optimum Moisture} = S\left(\frac{B}{A}\right) + K_2 \qquad (13)$$

LIMITATIONS

On the basis of the limited experimental work done so far, the modified calculation procedure developed in this study is sufficiently accurate to justify its use for calculating standard Proctor density and optimum moisture content in situations where a high degree of accuracy is not necessary. Where highly accurate results are required, the standard Proctor laboratory method should be used. The data obtained from experiments on eight soils in the Soils Laboratory at Iowa State University illustrate the results obtainable with the modified formulas (table I).

TABLE I. RESULTS OF PERSONAL LABORATORY EXPERIMENTS

Sample	1	2	3	4	5	6	7	8
Location	Iowa	Iowa	Iowa	Iowa	Iowa	Iowa	Iowa	Virginia
P.R. Classification	A-4	A-6	A-7-6	A-6	A-7-6	A-7-6	A-6	A-2-6
Mechanical Analysis, % Passing:								
No. 4 Sieve			99.8	99.2	100.0	82.8		47.0
10			99.0	98.0	99.9	73.6	100.0	42.0
20		100.0	96.6	95.1	99.8	66.1	99.6	37.1
40		99.9	93.0	89.2	98.9	59.0	97.0	32.3
60		99.9	87.3	79.6	97.3	52.9	94.9	24.5
80		99.8	83.4	73.5	96.1	49.1	93.6	.....
100		99.8	80.9	69.2	95.4	45.7	92.7	21.7
200	100.0	99.6	74.9	61.6	94.4	42.6	90.3	18.1
Soil Constants:								
Liquid Limit, %	29.5	34.8	51.0	30.7	66.0	41.0	38.1	31.5
Plastic Limit, %	20.0	19.7	20.7	12.8	27.0	19.1	23.9	19.8
Plasticity Index, %	9.5	15.1	30.3	17.9	39.0	21.9	14.2	11.7
Shrinkage Limit, %	18.9	15.9	9.8	11.0	10.8	8.9	11.4	14.9
Shrinkage Ratio	1.76	1.82	2.02	2.02	2.11	2.18	2.00	1.80
Standard Proctor								
Density, p.c.f.	107.6	110.0	106.6	120.0	98.2	124.1	117.2	119.8
Opt. Moisture, %	16.5	16.5	16.8	11.7	22.0	10.5	14.0	12.0
Calculated:								
Density, p.c.f.	107.5	107.2	108.6	119.1	103.2	127.1	118.9	118.5
Opt. Moisture, %	18.1	16.9	15.1	11.9	19.7	9.6	11.9	10.2

One of the greatest limitations of the modified calculation procedure is that it cannot be used with accuracy for organic soils and for mineral soils having a high organic matter content. Organic matter is highly absorptive, and its presence in the soil makes it extremely difficult to obtain accurate determinations of the Atterburg shrinkage and plasticity limits.

Another limitation is that the calculation procedure is sensitive to small changes in the shrinkage limit and the shrinkage ratio. Because of this, the shrinkage test should be performed with particular care. To obtain the best results with the formulas, the shrinkage factors used should be the average values of several shrinkage tests and should be determined to the nearest one-hundredth.

While much more research is necessary before the true value of the modified formulas can be established, the great savings in time and labor obtainable by the use of them justify their further study and development.

ADDENDA

D. T. Davidson

The extremely variable and complex nature of soils and the difficulties of moisture control make precise measurement of the maximum density impossible. Even with the widely used standard Proctor control test, it is generally impossible to obtain repeat values from a series of tests on the same soil. The Proctor density does, however, approximate the compaction obtainable by field equipment and experience has proved the test to be an invaluable aid in the construction of highways, airfields, and rolled earth dams. Extreme accuracy is not warranted for this determination because research has shown that there is no appreciable difference in watertightness or strength if the soil is compacted within two pounds of the Proctor density. For many engineering purposes not even this accuracy is needed since the majority of construction specifications require only that compaction be not less than 95 percent of the Proctor density.

During the summer and fall of 1949, two senior Civil Engineering students at Iowa State University made a statistical study under the supervision of Professor M. G. Spangler to compare the accuracy of the laboratory and calculation procedures for determining standard Proctor density. Only one soil, a sample of Mankato glacial till from Hamilton County, Iowa, was used in their study. It classified as A-6(8) by the revised BPR soil classification system.

To determine the degree of accuracy that could be obtained by the conventional standard Proctor laboratory test, the two students performed a series of 20 density tests on the single soil. Their results showed a maximum value of 116.1 p.c.f., a minimum value of 113.2 p.c.f., and an average value of 114.5 p.c.f. The small sample theory of the method of least squares was used to compute the plus or minus variance of the data with respect to the true mean. The analysis indicated that the laboratory test can be performed with an accuracy of $\pm$ 4 p.c.f. at least 99.7 percent of the time.

They next determined the number of shrinkage and plasticity index tests necessary to enable the standard Proctor density to be calculated by means of formula (8) with an accuracy of $\pm$ 4 p.c.f. This was accomplished by performing the shrinkage limit, shrinkage ratio, and plasticity index tests 20 times and then using the small sample theory method to calculate the number of each of these tests necessary to give the variance of $\pm$ 4 p.c.f. 99.7 percent of the time.

On the basis of their study, they concluded that the calculation procedure can be as accurate as the laboratory method if two plasticity index tests and five shrinkage tests are performed and the average values used in formula (8). Whether or not this conclusion is applicable to all soils will not be known until more studies of this nature are made on the various types of soil.

A VERSATILE RUBBER BALLOON APPARATUS FOR MEASURING IN-PLACE DENSITIES OF SOILS

by

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(Highway Research Board Bulletin, 122. 1955.)

ABSTRACT

In-place densities of soils are usually determined by augering or digging a hole, weighing and determining the moisture content of soil from the hole, and measuring the volume of the hole. The volume may be measured by the oil method, by the sand-cone method, or by using a rubber balloon apparatus. The rubber balloon method is quite accurate.

Various kinds of commercially available soil density apparatus utilizing a rubber balloon have been designed especially for measurements of densities of sub-grades, bases, and compacted earth fills. These are limited to use on fairly level surfaces. Work in Iowa on natural soils showed a need for an apparatus which could be used either on level, sloping, or vertical faces. With such an apparatus, measurements could be made in roadcuts, in quarry faces, in borrow pits, in basement excavations, and in other like places deep in the soil section. Additional requirements for the design of the apparatus were that it be rugged enough to withstand rouge field usage, and light and compact enough for use by a man suspended from a rope over a roadcut or quarry wall. A new rubber balloon apparatus was developed to meet these requirements. As a result of two years of use of the new apparatus in Iowa and Alaska, various modifications and improvements have been made.

INTRODUCTION

During the past several years, soils investigations by the Iowa Engi neering Experiment Station have included in-place density measurements at a large number of locations in and out of the state. It was often desired to extend these measurements to depths of tens of feet in the soil materials, and quarries and roadcuts became almost a necessary convenience. However, the common disturbed methods of density measurement—the oil, the

sand-cone, and the rubber balloon methods[1, 2, 3, 4]—are all adapted to measurements on a level surface. On a vertical face or a steeply sloping face, this required an undesirable amount of hand excavation; therefore a new apparatus was designed. The desirable features in the new apparatus were that it be equally adapted for use on level, sloping, or vertical faces, and that it be light and handy enough to be operated by one person hanging on a rope swing.

A modified rubber balloon apparatus was designed and constructed to meet these requirements. During the trial and development period of two years, many modifications have been made, and some rather unique features have been incorporated. The apparatus has been used and tried in various soils in Iowa and Alaska. It is now used by the Experiment Station for all field density tests, including those on level surfaces.

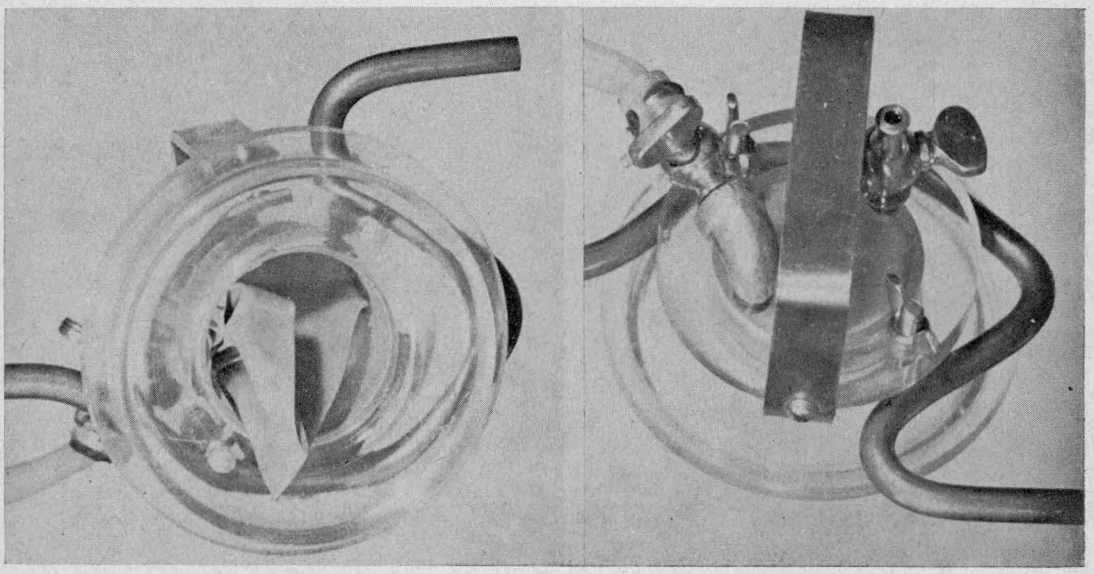

Fig. 1. The balloon holder clipped into the steel cylinder support for carrying. In the left photograph may be seen the concave, rounded bottom of the balloon holder designed to minimize balloon breakage. The balloon is drawn inside of the holder as for a rapid zero reading.

Tests are now being conducted with disposable polyethylene plastic sample bags. Since the plastic is waterproof, the soil can be placed in bags

which then are sealed and returned to the laboratory for weighing and moisture content determinations. This eliminates the need for transporting a delicate weighing mechanism into the field. The plastic bags cost about ten cents apiece, and are reusable.

The Iowa balloon apparatus is essentially a folding template and a small balloon holder, the holder being connected by a 3/8 inch plastic hose to a 3 foot cylinder graduated in 0.001 cubic foot increments (figure 1). The testing operation is as follows:

> A smooth face is cut on the soil. The sample bag is put in place on the template and the template is pushed against the smoothed face, where it is held by pins projecting from the back of the template into the soil. With the hole in the template as a guide, a hole 4 inches in diameter and 4 to 6 inches deep is cut in the soil. On steep faces, all soil falls into the sample bag, and the bag can be removed and weighed immediately. On level or near level surfaces, the excavated soil is pushed away from the hole in the template; the balloon holder is fitted into this hole, and the volume of the excavation is measured. One of the flow valves is turned off, the balloon is removed from the hole, and the sample bag is then removed and weighed. A moisture determination sample is taken from soil in the sample bag. Zero measurements are required for volume, the weight of the moisture can, and the weight of the sample bag. (A detailed procedure is given in Appendix I.)

Fig. 2. For a rapid zero check, pressure is applied to draw the balloon up into the holder. A flow valve is turned, and the graduated cylinder is held vertically while the water level is read.

Fig. 3. Apparatus assembled for carrying. The balloon holder and loose hose fasten onto the graduated cylinder support, and (right) the folding template fits into the sample bag for carrying.

Unique Features and Advantages

Incorporated into the apparatus are several features believed to be advantageous:

1. The graduated cylinder, balloon holder and template are all constructed of plexiglass—easy to see through but hard to break. Breaks can be repaired with common household cements, most of which have a solvent effect on the plastic.

2. The balloon holder is concave and rounded on the bottom, minimizing balloon breakage (figure 1).

3. Water flow is obtained by changing the head, so pumps or suction devices are unnecessary. For example, after running a test a flow valve is turned off, and the water is returned from the balloon to the cylinder by removing the balloon from the hole and holding it higher than the cylinder. The flow valves are opened, and the water flows back into the graduated cylinder.

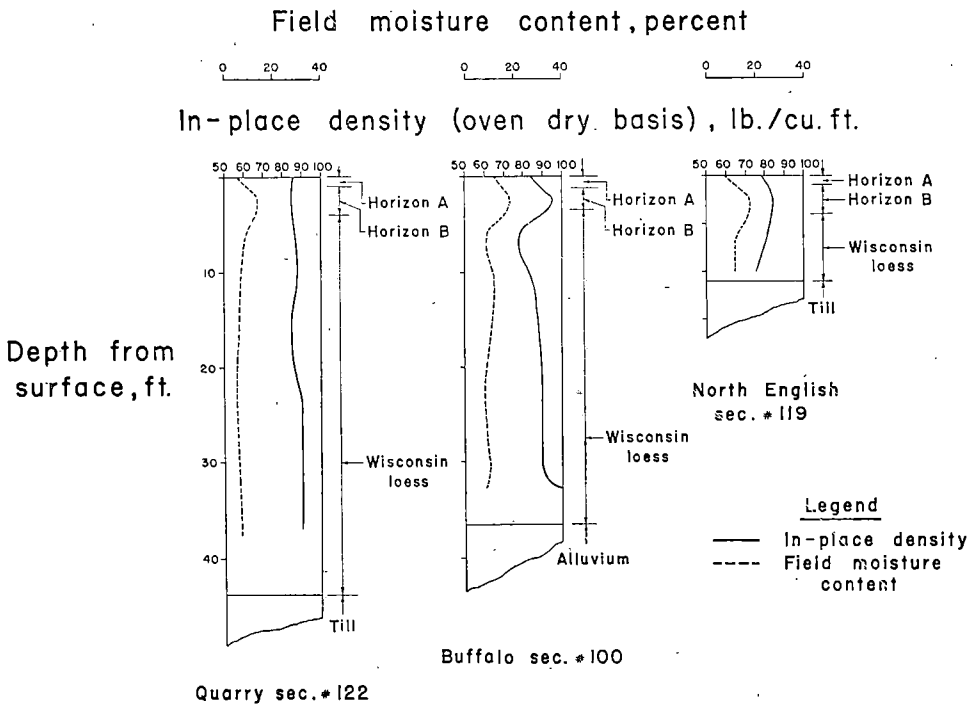

Fig. 4. Depth field density relationships obtained with the Iowa apparatus. Measurements were made in roadcuts and quarries.

4. A rapid check of the zero reading to determine effects of temperature changes during the day can be made by raising the balloon holder so that the water drains into the cylinder (figure 3). The balloon is thus drawn up tightly inside the balloon holder, giving a base point for a zero reading (figure 2). This reading must be calibrated to the normal zero reading obtained by inflating the balloon with the holder against a flat surface.

5. To secure complete inflation of the balloon inside the hole, pressure is applied by locating the cylinder above the balloon holder. If necessary, additional pressure can be applied by blowing into a hose connected to the top of the cylinder.

6. The apparatus uses ordinary toy balloons with the necks cut off. Balloons are replaced by dismantling the balloon holder with two thumb nuts. The balloon itself forms the gasket.

7. The apparatus clips together into a balanced unit which can be lowered on a rope or carried in one hand (figure 4).

Precautions and Disadvantages

1. Due to leaks or balloon changes, air may get into the balloon holder. To remove the air water is run into the balloon, the holder air valve is opened, and the air is forced out by squeezing the balloon. The valve is then closed.

2. Reasonable care must be taken during the test to prevent kinks in the hose. If there are no kinks, the plastic hose holds its shape well and does not expand appreciably with the pressures used, and the volume reading is not changed by moving the hose.

3. The sample bag must be shaken out and re-weighed prior to every test.

4. The mouth of the sample bag may stretch so that it does not fit the template tightly. To correct this, the drawstring is drawn shorter and tied.

5. The steel rod is forced into the ground to support the graduated cylinder. (A foot rest is provided.)

6. The overall length of the apparatus is about five feet, and care must be used to prevent breakage in transportation.

7. The calibrated cylinder is two inches in internal diameter and has a capacity of 0.06 cubic foot. Therefore the maximum depth for a four inch hole in the soil is about 7½ inches. If desired, a larger cylinder could be used, but the smaller size was found to be satisfactory for most soils, and superior from the standpoint of portability.

APPLICATIONS OF THE APPARATUS

The new balloon apparatus, in addition to being used for ordinary borrow or compaction operations, may be used to determine densities through

deep sections in soils (figure 4). These data show that the in-place density of loess gradually increases with increasing depth, probably due to the weight of the overlaying material. Quite often the loess increases greatly in density near the basal contact, probably due to mixing with the underlying material and to puddling by the water table.

Incidental to the development of the density apparatus, a rope swing was used in sampling and testing high, steep faces. The apparatus consists of a board seat suspended by a three-eighths or one-half inch rope on a 3 to 1 block and tackle with the double block at the top. The top block is suspended from an eye at the end of a 6 foot collasible aluminum beam. The beam was fabricated from 2 inch angles, and has a bearing plate 1½ feet back from the outer end. The other end is fastened to a corkscrew type soil anchor. A safety rope is an accessory.

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4. Minor, C. E., and Humphres, H. W. A new method for measing in-place density of soils and granular material. Hwy. Res. Bd. Bulletin 93. 1954.
5. Rainhart Company, Austin, Texas, Instructions for use of the Rainhart rubber balloon apparatus.

APPENDIX I

SUGGESTED PROCEDURE FOR USE OF THE IOWA DENSITY APPARATUS

Apparatus

1. Iowa density apparatus, including calibrated cylinder, rubber balloon holder, template and sample bag.
2. Digging tools. A heavy kitchen spoon, hunting knife, chisel, prospector's pick, small pointed trowel, and a small flat shovel will be useful.
3. Tin cans with press tops for moisture tests. Four or eight ounce cans are satisfactory.
4. A balance of 3000 gram capacity, accurate to 0.1 gram in the lower range.
5. A stove or oven for drying samples. The alcohol burning procedure is an alternative method for drying[5].

Testing

1. Prior to testing, an average zero volume reading is obtained by inflating the balloon with the holder pressed against a flat surface (figure 1). The zero reading will ordinarily change only because of leaks or large temperature changes, but it should be checked occasionally. The check can be made by either repeating the zeroing operation or more conveniently by deflating the balloon so that it is drawn tightly back into the balloon holder. This is done by opening the flow valves and raising the holder four or five feet above the cylinder (figure 2). The zero thus obtained must be corrected by adding to it the volume of the balloon holder. This volume can be determined by following the above procedure at the time of the initial zeroing and comparing the two zero readings.

2. The sample bag is shaken out and weighed prior to each test. To remove loose soil, the sample bag can be turned inside out, shaken and used that way in the next test.

3. The soil is shaved and smoothed off to a flat surface at the site of the test. The area should be about one foot square and may be inclined. A shallow groove is scraped across the lower part of the area to make room for the seam of the sample bag.

4. The cylinder support is pushed into the ground near the test site to hold the cylinder vertical. The balloon holder is unclipped and set aside ready for use.

5. The folding template is removed from the sample bag and enough pins installed to hold it to the soil. Unless the soil is loose, three of the short pins, two at the top, will usually suffice.

6. The sample bag is placed on the folding template and the template braced open.

7. The assembled template is pushed against the soil face (figure 1); the pins will be pushed into the soil and hold the template there. If the template does not fit solidly, it should be removed and the soil face trimmed. The plexiglass allows one to see irregular contacts.

8. The test hole is dug through the hole in the template, and the excavated soil placed in the sample bag and/or on the template (figure 1b). The hole should be smooth and approximately four to six inches deep.

9. (a) On steep slopes all excavated soil falls into the sample bag, and the bag can be removed and weighed immediately. (b) On level or near level surfaces the bag is left in place, the soil is pushed away from the hole in the template, and the balloon holder installed (figure 1). The flow valves are opened; the balloon fills with water and expands into the test hole. The balloon holder must be held down with a hand, knee or foot. Additional water pressure can be applied by either raising the cylinder or blowing in the cylinder air hose. As more pressure is applied, it should be noted if there is any increase in the reading. An increase would indicate that the balloon does not yet completely fill the hole, and the higher pressures are necessary. Caution must be used lest the balloon holder be lifted off the template. A six foot water head obtained by raising the cylinder will exert about 2.5 psi pressure in the balloon, and the balloon holder must then be held down with a force of about 30 pounds.

10. When the balloon is inflated to a maximum inside the hole, one of the flow valves is turned off, and the balloon and holder are lifted from the hole and set aside. The volume can be read and recorded immediately or after the sample sack has been removed and weighed. If the operator is working alone, the latter procedure is advisable to reduce evaporation from the soil sample.

11. The template is lifted and the soil is brushed into the sample bag, which is then removed and weighed. A moisture can is filled with soil from the middle of the sample bag and the weight of the filled can is recorded. The soil in the can is later dried in an oven or by the alcohol burning method and the moisture content calculated.

12. The volume reading is estimated to 0.0001 cubic foot. The flow valves are opened and the balloon held above the cylinder so that the water flows back into the cylinder. The operation can be speeded up by squeezing the balloon.

13. All valves are closed and the apparatus either folded up or made ready for another test. Calculations of moisture content and dry density are illustrated in Appendix II.

Special Procedures

14. *Removing air from the system.* This is seldom necessary unless there are leaks. The flow valves are opened and some water is allowed to run into the balloon. A flow valve is shut, the air valve on the balloon holder is opened, and the air is forced out by squeezing the balloon. It is necessary to re-zero after this operation.

15. *Replacing a broken balloon.* As much water as can be saved is run back into the cylinder, and more water added if necessary. Then the flow valves are closed and the balloon holder is disassembled by removing the two wing nuts. A new balloon is installed smoothly over the end of the holder tube and the holder reassembled. Air is removed (Procedure 14), and the apparatus re-zeroed. Ordinary round toy balloons are satisfactory for the test, although large sizes may be preferable. The neck is cut from the balloon at a point where it is somewhat smaller than the tube of the balloon holder.

16. *Filling or adding water to the apparatus.* If a water tap and small hose are available, water can be introduced at either the cylinder air valve or the balloon holder air valve. In either case all valves are opened and the apparatus arranged to allow air to escape. If no hose or water pressure is available, the cylinder can be filled by removing the balloon and pouring water into the upturned balloon holder. The flow valves and the cylinder air valve must be open. The balloone is then replaced (Procedure 15), the air removed, and the apparatus re-zeroed.

APPENDIX II

SAMPLE DATA AND CALCULATION SHEET

Location: F1A-1, Fairbanks, Alaska.
Material: Very dry, buff-colored friable silt.
Compaction: None.
Depth of Test: 2′7″ to 2′11″ (vertical roadcut)

Hole:		
	Final reading	0.0336 cu. ft.
	Zero reading	0.0171 cu. ft.
	Hole volume	0.0165 cu. ft.

Moisture Content:

Wt. can + moist soil	149.8 gm.	Wt. can + dry soil	144.3 gm.
Wt. can + dry soil	144.3 gm.	Wt. can	30.0 gm.
Wt. moisture	5.5 gm.	Wt. dry soil	114.3 gm.

$$\text{Moisture Content} = \frac{5.5}{114.3}(100) = 4.8\%$$

Density:

Wt. sack + moist soil	729.5 gm.
Wt. sack	40.6 gm.
Wt. moist soil	688.9 gm.

$$\text{Wt. dry soil} = \frac{688.9}{100 + 4.8}(100) = 657 \text{ gm.} = 1.45 \text{ lb.}$$

$$\text{Dry density} = \frac{1.45 \text{ lb.}}{0.0165 \text{ cu. ft.}} = 87.9 \text{ lb/cu. ft.}$$

CATION EXCHANGE CAPACITY

OF LOESS AND ITS RELATION TO ENGINEERING PROPERTIES

by

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J. B. Sheeler, Associate Professor, Civil Engineering

(ASTM Special Technical Publication, 142:1-19. 1952.)

ABSTRACT

Cation exchange capacity of the "whole" soil was one of the properties measured in an investigation of the variation of physical and chemical properties of the Wisconsin loess in southwestern Iowa. The test data of the property variation study affords an unusual opportunity for correlations of cation exchange capacity with some engineering (physical) properties of a natural soil material.

WISCONSIN LOESS OF SOUTHWESTERN IOWA

Origin

The Wisconsin loess (also called Peorian loess) which blankets older (pre-Wisconsin) loesses and glacial deposits in southwestern Iowa is believed to have accumulated during and immediately following glaciations of the Wisconsin glacial stage which invaded northern Iowa and Nebraska. Four glacial drifts in northwestern Iowa of Wisconsin age, the Iowa, Tazewell, Cary, and Mankato, have recently been mapped. Most geologists now agree that the Wisconsin loess in southwestern Iowa was deposited by the wind. The major source of this loess appears to have been the flood plain of the Missouri River, but undoubtedly the flood plains of other outwash carrying valleys of the region also contributed. Some loess was also blown directly from the Wisconsin drift plains in northwestern Iowa.

Distribution

The topography of the Wisconsin loess area included in the investigation has been described as loess depositional and loess mantled erosional[11].

Most of the depositional topography is in a narrow strip three to twenty miles wide bordering the flood plain of the Missouri River; to the east is the loess mantled erosional topography. Another strip of depositional topography, one to two miles in width, is immediately south of the Wisconsin (or Iowan) drift border[15]. The characteristic features of the loess depositional topography are the narrow divides and the steep "cat-stepped" slopes which

are cut in many places along valley walls by U-shaped gullies. The relief is often more than 200 feet within distances of a few miles.

In the loess mantled erosional topography, which grades into the loess depositional topography to the north and west, the modifications of the old pre-Wisconsin erosion slopes take the form of rolling hills, with the hills becoming more gently rolling with increasing distance away from the Missouri River flood plain. The loess is for the most part on the tops of hills and on the upper parts of slopes. Thinner loess extends into and across the valleys.

The thicknesses of Wisconsin loess in the loess depositional topography are greater than in the loess mantled erosional topography. For example, the maximum thicknesses of the loess, as measured on ridges and hilltops, vary from 60 to over 100 feet along the Missouri River bluffs to about seventeen feet at the arbitrary east boundary (figure 1). The loess thins in a southeasterly direction away from the valley of the Missouri River (figure 2). The depth measurements plotted in the graph include the thickness of the A and B horizons where both are present. The principal soil association areas in the Wisconsin loess area under study are the Monona-Ida-Hamburg and the Marshall[14]. The thicknesses of the solum on ridges and hilltops vary from zero or a few inches in depth at the west boundary to around three or four feet near the east. Solum includes both the A and B soil horizons where both are in place or one only where the other is missing.

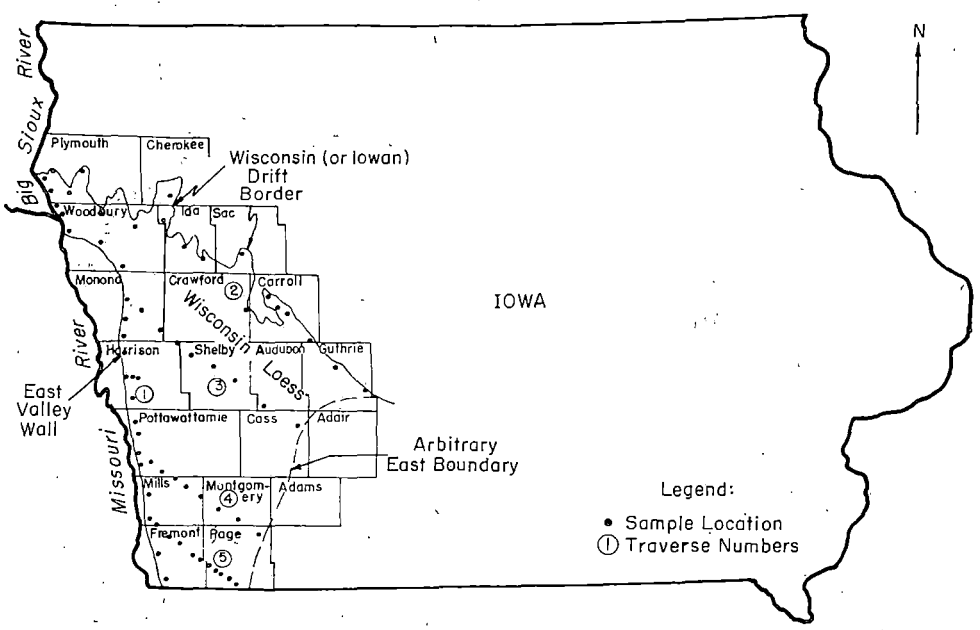

Fig. 1. Locations of sampling traverses in Wisconsin loess area of south western Iowa.

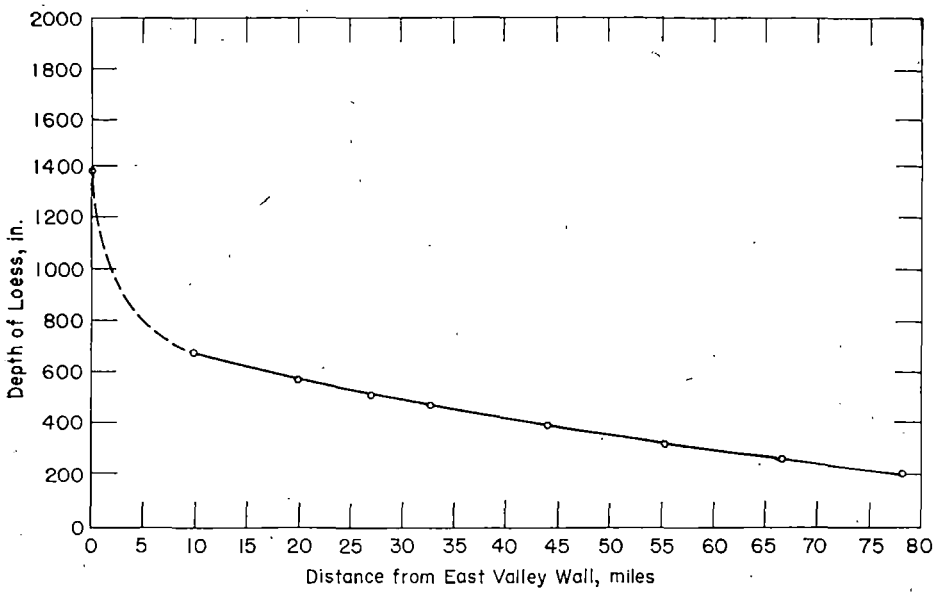

Fig. 2. Variation in maximum thickness of Wisconsin loess along traverse 3, as interpolated from Hutton's data[10]. The depth measurement at the east valley wall is the estimated maximum thickness of the loess in the general vicinity of the traverse origin.

Properties

Over one hundred samples of Wisconsin loess have been taken along the five traverses (figure 1). Traverse 1 is along the top of the east valley wall of the Missouri River and, for a short distance, along the Big Sioux River. Traverse 2 is just south of the Wisconsin drift border. Traverses 3, 4, and 5 were laid out in a southeasterly direction, which is believed to represent with a reasonable degree of accuracy the direction of the generally prevailing winds during loess deposition time.

Control samples for determining areal property variations were taken at a depth of from two to three feet below the top of the C horizon at each of the locations shown on the map; at many of the locations samples were also taken at greater depths for the purposes of the stratigraphic variation study. No samples were taken from the A or B horizons. All sampling was done on ridges or hilltops. A 6 inch diameter soil auger was used for securing samples when suitable road cuts could not be found.

The tests which are being used to determine the areal and stratigraphic variations in the properties of the Wisconsin loess are as follows:

1. Liquid limit (L.L.)
2. Plastic limit (P.L.)
3. Plasticity index (P.I.)
4. Shrinkage limit (S.L.)
5. Centrifuge moisture equivalent (C.M.E.)
6. Field moisture equivalent (F.M.E.)
7. Hygroscopic moisture (air dry)
8. Mechanical analysis
9. Specific gravity
10. Field moisture content
11. In-place (field) density
12. Wet and dry color
13. Textural and engineering classification
14. Hydrogen ion concentration, or pH
15. Organic matter content
16. Carbonate content, expressed as percent $CaCO_3$
17. Sulfate content, expressed as percent SO_3
18. Cation exchange capacity
19. Differential thermal analysis
20. Types of exchangeable cations

The following discussion of properties of the Wisconsin loess is made on the basis of available test results. Wisconsin loess in the southwestern Iowa area is a fine textured, predominantly silt and clay size soil material. The dry Munsell color of the oxidized loess is pale yellow, light yellow brown or light olive brown. The unoxidized loess is light gray. Near its source where it is deepest, the loess is commonly highly calcareous. With increasing distance away from its source, the loess becomes thinner, finer textured, more weathered, and less calcareous. Wisconsin loess shows essentially no stratification and a characteristic feature is its ability to stand in vertical or near vertical slopes[7]. Striking examples of this feature may be seen in many cut sections in the bluffs along the Missouri River (figure 3).

The test data for traverse 1 indicate that physical and chemical properties of the Wisconsin loess along the east valley wall of the Missouri River are remarkably uniform both areally and stratigraphically, for a natural

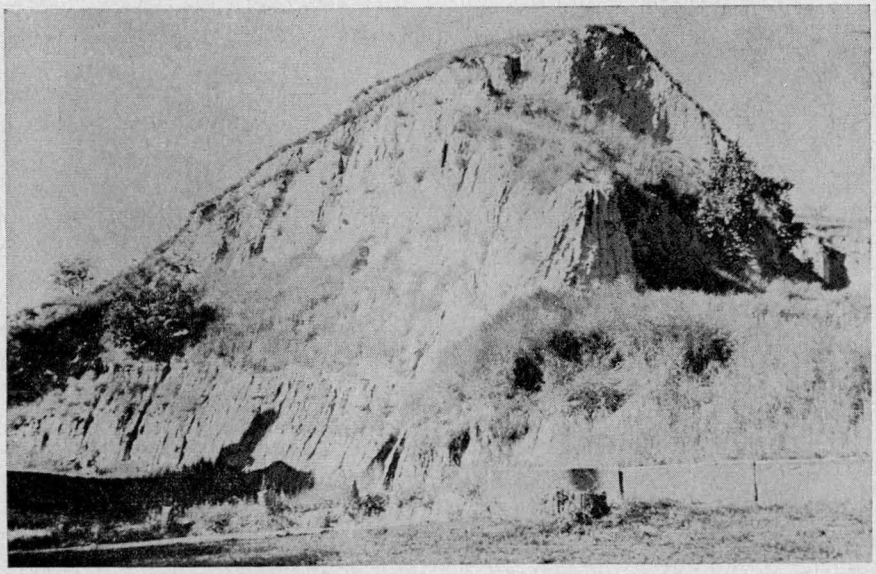

Fig. 3. Exposure of Wisconsin loess in the east valley wall along the Missouri River behind the third ward school at Missouri Valley, Harrison County, Iowa. This cut shows 75 feet of Wisconsin loess overlying 15 feet of pre-Wisconsin (Loveland) loess.

deposit of soil material. As would be expected, in-place density increases with depth in the loess; the field moisture content also shows some increase with depth.

With increasing distance away from the east valley wall, however, test data for samples taken along traverses 3, 4, and 5 reflect a marked increase in plasticity, shrinkage, water-holding capacity, and in-place density; in general, the data of traverse 2 shows the same trends. These property changes appear to be chiefly due to an increase in the amount of clay in the loess[6], the textural classification of the loess changing from silty loam at the west boundary to silty clay at the east. With the exception of increase of field moisture content and in-place density, the values of the above mentioned properties are comparatively uniform at varying depths in the loess.

The mineralogical nature of Wisconsin loess in the southwestern Iowa area seems to be quite uniform. Organic matter and soluble sulfate contents are low or nonexistent. Aside from variation in amount of clay, the principal variable in the loess appears to be carbonate content, which varies both areally and stratigraphically. Carbonate percentages, expressed as percent calcium carbonate by weight, were as high as eighteen percent along the east valley wall of the Missouri River and decreased to as low as one percent in samples obtained near the arbitrary east boundary.

TABLE I. PROPERTY VARIATIONS IN WISCONSIN LOESS ALONG TRAVERSE 3

Sample	County	Soil Series	Distance from Valley Wall, miles	Depth* or Loess, in.	Sampling† Depth, in.	Physical Tests				Chemical Tests				Field Tests		Textural Composition, percent§			Textural classification (B.P.R. System)	Engineers Classification (B.P.R.)
						P.I. percent	C.M.E. percent	S.L., percent	Sp. Gr., 25 C/4 C	pH	Org. Mat., percent	Carbonates, percent	Cat. Ex. Cap. (m.e./100 g)	Field Moist., percent	In-Place‡ Density lb. per cu. ft.	Sand	Silt	Clay		
No. 22-1	Monona	Hamburg	0	1400**	24-36	5.7	11.7	24.7	2.70	8.6	0.30	15.0	10.4	6.8	69.4	2.2	82.2	15.6	Silty Loam	A-4 (8)
No. 23-1	Monona	Ida	9.8	690	24-36	5.3	14.8	24.2	2.71	8.6	0.22	12.3	14.7	.....	.....	2.2	81.2	16.6	Silty Loam	A-4 (8)
No. 24-1	Monona	Ida	20.0	590	24-36	5.2	19.3	22.4	2.71	8.4	0.34	11.8	14.3	13.8	73.5	2.1	75.1	22.8		A-4 (8)
No. 24-2	Monona	Ida	20.0	590	348-360	5.5	20.0	22.1	2.71	8.6	0.16	9.8	14.6	20.8	89.5	1.3	75.0	23.7		A-4 (8)
No. 25-1	Harrison	Monona	27.0	525	24-36	14.4	20.1	22.3	2.71	8.3	0.40	12.6	15.5	.....	.....	1.7	70.5	27.8	Silty Clay Loam	A-6 (10)
No. 25-2	Harrison	Monona	27.0	525	90-102	12.1	18.9	22.4	2.70	8.3	0.20	10.0	15.7	.....	.....	1.7	73.5	24.8		A-6 (9)
No. 26-1	Shelby	Monona	32.7	485	24-36	12.5	19.5	23.3	2.70	7.0	0.18	1.4	18.2	22.9	76.2	2.0	70.6	27.4		A-6 (9)
No. 26-2	Shelby	Monona	32.7	485	96-108	17.8	21.6	21.9	2.69	8.3	0.17	8.7	17.9	25.5	87.4	0.9	69.8	29.3		A-6 (9)
No. 27-1	Shelby	Marshall	44.0	405	24-36	18.2	22.1	21.3	2.70	7.0	0.16	1.5	19.3	.....	.....	0.9	68.1	31.0	Silty Clay	A-7-6 (12)
No. 28-1	Shelby	Marshall	55.3	335	24-36	16.2	20.8	22.0	2.70	8.4	0.21	7.6	17.6	27.7	79.6	1.2	69.1	29.7	Silty Clay Loam	A-6 (10)
No. 29-1	Audubon	Marshall	66.6	275	24-36	18.0	21.5	18.9	2.70	8.3	0.25	2.9	19.5	.....	.....	1.0	67.9	31.1	Silty Clay	A-6 (11)
No. 30-1	Cass	Marshall	78.2	220	24-36	26.6	25.4	17.8	2.70	6.9	0.21	1.6	20.4	28.0	83.5	1.1	60.2	38.7	Silty Clay	A-7-6 (16)

* Depth measured from earth's surface.
† Sampling depth refers to that measured from the top of C-horizon.
‡ In-place density is expressed as dry density.
§ Sand—2.0 to 0.05 mm., silt—0.05 to 0.005 mm, clay—below 0.005 mm.
**Estimated.

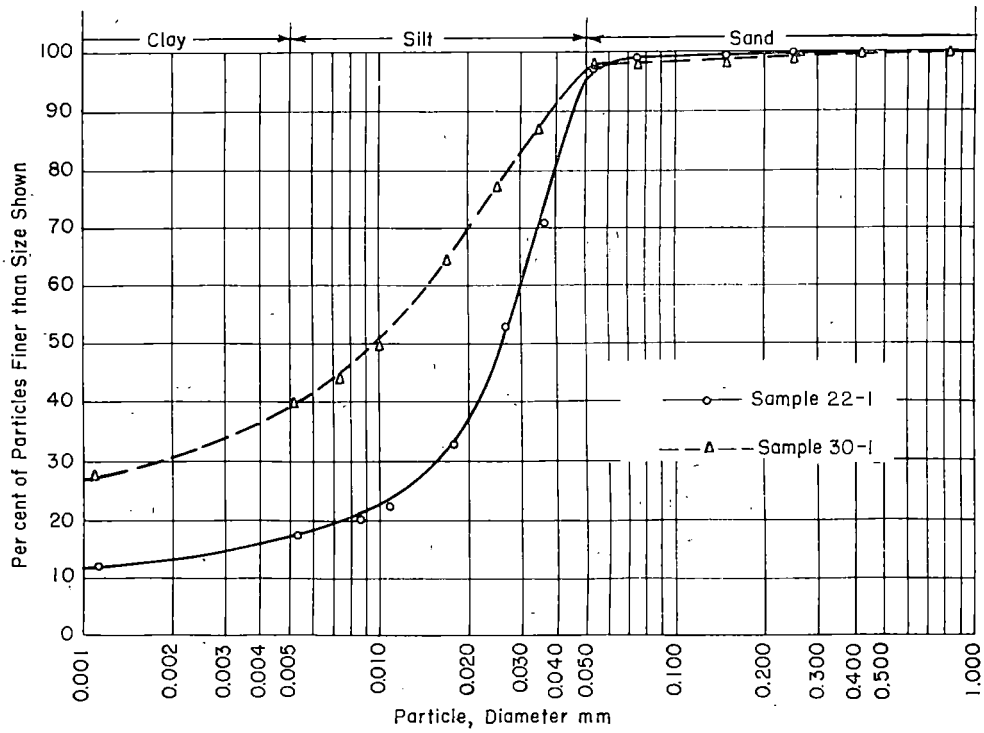

Fig. 4. Particle size accumulation curves for samples obtained at west (22-1) and east (30-1) ends of traverse 3.

Part of the above discussion pertaining to results of the property variation study is illustrated by table I, which shows the trends of property data for the loess samples taken along traverse 3. Figure 4 shows particle size accumulation curves for samples obtained at the west and east ends of the same traverse (samples 22-1 and 30-1 in table I). Wisconsin loess in the southwestern Iowa area is classified into Bureau of Public Roads system groups[2] (figure 5) ; the Bureau of Public Roads group boundaries as shown should be considered as approximate.

CATION EXCHANGE CAPACITY IN WISCONSIN LOESS

Cation exchange capacity is one of the most important properties of soil, a fact that has long been recognized by agricultural soil scientists[12]. It can be measured qualitatively and is closely related to the physical and chemical behavior of soil.

A knowledge of the cation exchange capacity is essential for the proper treatment of engineering soils with such chemical admixtures as large organic cations[4] and lime[3]. In studies of the clay fraction of engineering

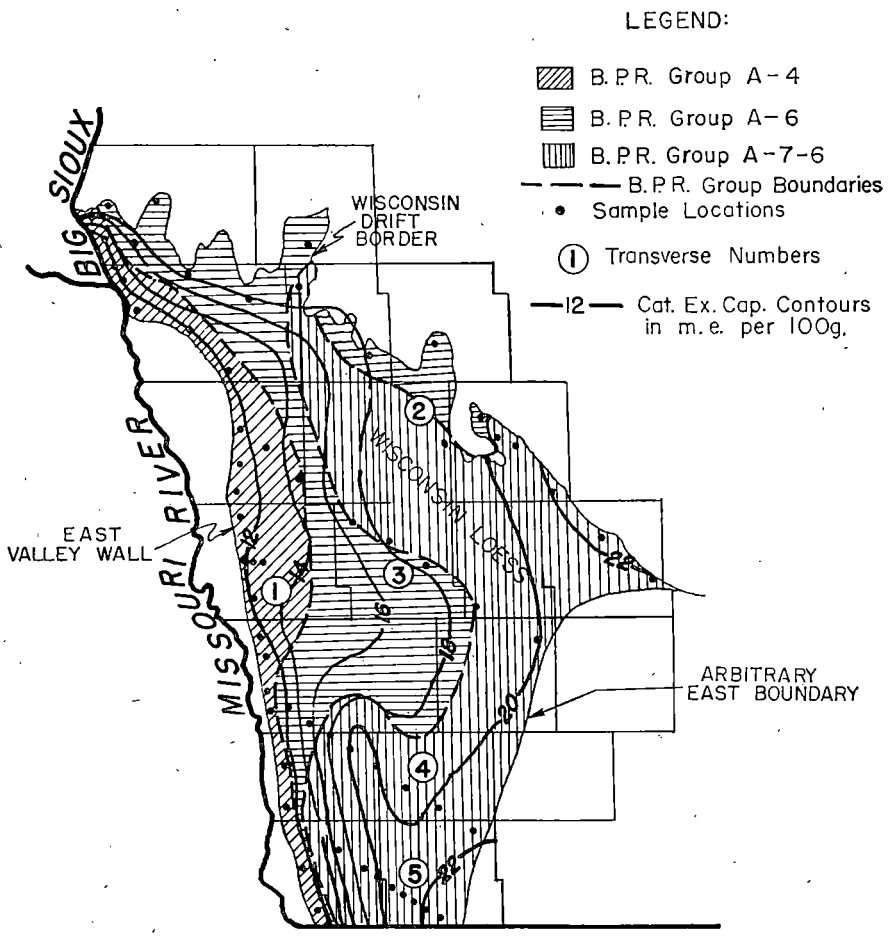

Fig. 5. Wisconsin loess area in southwestern Iowa showing classification of loess by BPR system, and variation of cation exchange capacity of loess by contours spaced at 2 m.e. per 100 g intervals.

soils, the cation exchange capacity of the extracted clay colloids often can be used as a guide in identifying the predominant kind of clay mineral present.

The extent to which different soils are able to exchange cations with solutions varies widely. Generally speaking, the exchange capacity of engineering soils depends upon two variables, the amount and the kinds of clay minerals present. Where kind of clay mineral is the principal variable, soils containing montmorillonite minerals are relatively high in exchange capacity; those in which illite is the predominant kind of clay mineral are intermediate, while kaolinite soils are usualy low in exchange capacity.

In Wisconsin loess the organic matter content is low (table I), and the inorganic clay minerals may be considered the primary seat of cation exchange. The clay mineral composition of the loess appears to be uniform. Studies to date indicate that minerals of the illite group predominate, but very likely some montmorillonite group minerals are also present. Calcium appears to be the principal exchangeable cation associated with the clay material. Further studies are now in progress to determine more definitely the mineralogy of the Wisconsin loess in southwestern Iowa.

Cation exchange capacity is usually expressed in milliequivalents of cation per 100 grams of oven-dry soil (m.e. per 100 g). A milliequivalent may be defined as one milligram of hydrogen (H+) or the amount of any other cation that will displace it. Other cations may be expressed in milliequivalents by changing them over into their hydrogen equivalents. If the cation exchange capacity of a soil is known, the grams of any particular cation that it can adsorb may be calculated as follows:

$$\text{grams adsorbed per 100 g of soil} = \text{m.e. per 100 g} \times \frac{\text{atomic wt. of cation}}{\text{valence of cation} \times 1000}$$

For example, a soil having a cation exchange capacity of 20 m.e. per 100 g is capable of adsorbing

$$20 \times \frac{1.008}{1 \times 1000} = 0.0202 \text{ g of } H+,$$

$$20 \times \frac{40.08}{2 \times 1000} = 0.4008 \text{ g of } Ca++,$$

or

$$20 \times \frac{17.03}{1 \times 1000} = 0.3406 \text{ g of } NH_4+$$

per 100 g of oven dry soil.

Determination of Cation Exchange Capacity

Numerous methods for determining cation exchange capacity, many of which differ but slightly, have been proposed[12]. Most of them involve washing or leaching the soil sample with a salt solution containing known cations, followed by analysis of either the resulting soil or leachate for the amount of cations exchanged. It has been recognized for some time that the exchange capacity of a soil denotes the total amount of cations that can be exchanged under a given set of conditions and not necessarily the amount that could be exchanged under other conditions. The determination is particularly sensitive to the pH of the salt solution used in the leaching operation; with a neutral (pH = 7) solution of a given salt, the cation exchange capacity may be significantly less than when determined with an alkaline (pH>7) solution of the same salt. For this reason, the pH of the leaching solution should always be stated. The common practice is to use neutral solutions.

Neutral normal ammonium acetate is especially well adapted to the cation exchange capacity determination and was used in the Wisconsin loess study. With this ammonium acetate solution, the exchange capacity of engineering soils can be determined with reasonable accuracy even when the soil contains soluble salts and calcium carbonate. High accuracy depends upon thorough replacement of the cations in the soil with ammonium ions and then accurate determination of ammonium (NH_4+) that has been taken up by exchange. The test methods used in measuring the cation exchange capacity of the Wisconsin loess samples are presented in the appendix.

Cation Exchange Capacity of Wisconsin Loess

On the basis of tests on over one hundred samples of Wisconsin loess, the range in cation exchange capacity values in the southwestern Iowa area studied is from 7.5 to 25.8 m.e. per 100 g. The exchange capacity of the loess is lowest near the major source of the loess, the flood plain of the Missouri River, and increases in value with distance eastward away from the source. This increase is mainly attributed to an increase in the amount of clay in the loess. There seems to be very little stratigraphic variation of cation exchange capacity.

The above mentioned trends are illustrated in table I and figure 6 by the data for samples taken along traverse 3. In figure 5 the variation of cation exchange capacity in the southwestern Iowa area is shown by contours having a 2 m.e. per 100 g interval.

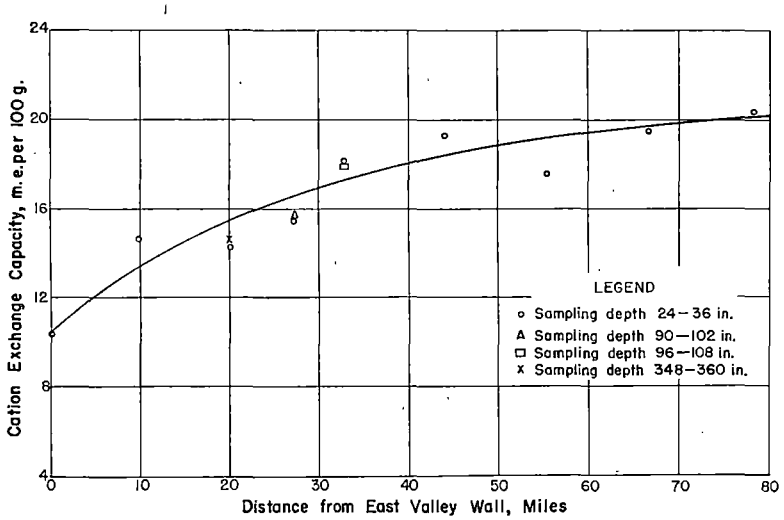

Fig. 6. Variation of cation exchange capacity of Wisconsin loess along traverse 3.

CORRELATION OF CATION EXCHANGE CAPACITY WITH ENGINEERING PROPERTIES

The test data for Wisconsin loess affords an opportunity for correlations of cation exchange capacity with some engineering properties which are commonly used for the identification, design, and construction control of soils and soil mixtures. Data available at the present time indicate that the major variables in the loess are the amounts of clay and carbonates present.

Properties Correlated

The engineering properties to be correlated with cation exchange capacity were determined by means of the following test procedures:

1. Percent 0.002 mm. clay (ASTM Method D 422-51, as modified[5])
2. Liquid limit (ASTM Designation: D 424-39)
3. Plastic limit (ASTM Designation: D 424-39)
4. Plasticity index (ASTM Designation: D 424-39)
5. Shrinkage limit (ASTM Designation: D 427-39)
6. Centrifuge moisture equivalent (ASTM Designation: D 425-39)
7. Field moisture equivalent (ASTM Designation: D 426-39)
8. Hygroscopic (air-dry) moisture (ASTM Designation: D 422-39)
9. In-place (field) density (the rubber balloon method was used)[8]
10. Field moisture content (The method for material that does not contain aggregate larger than 0.25 inch was used[8].)

Complete details on the tests may be obtained from publications cited[1, 9, 16].

Correlations

The relation of cation exchange capacity to the several engineering properties was determined by plotting, on linear graph paper, the value of the engineering property of each loess sample against the sample's cation exchange capacity (figures 7 to 16). The curves were visually fitted to the plotted points; no attempt was made to fit a curve to data in figure 13, which shows the relationship of the cation exchange capacity to the field moisture equivalent data. The in-place density points in figure 15 represent only the loess densities as measured at a depth of two to three feet below the top of the C horizon. This was necessary because in-place density shows

a marked increase with depth in the loess and cation exchange capacity does not. The plotted points in all other graphs represent Wisconsin loess at the two to three foot depth or deeper.

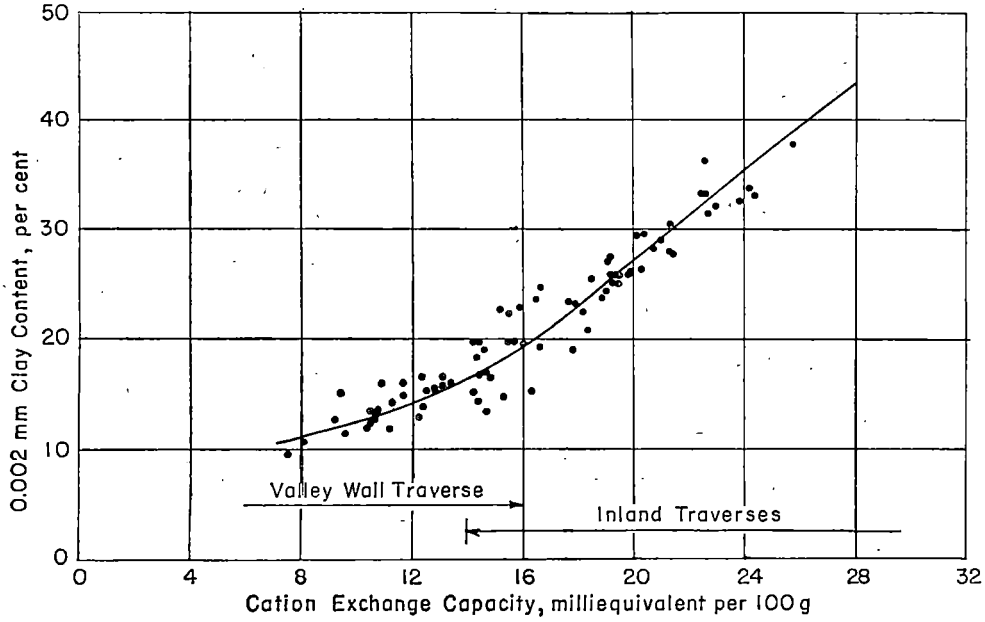

Fig. 7. Relation of cation exchange capacity to 0.002 mm. clay content.

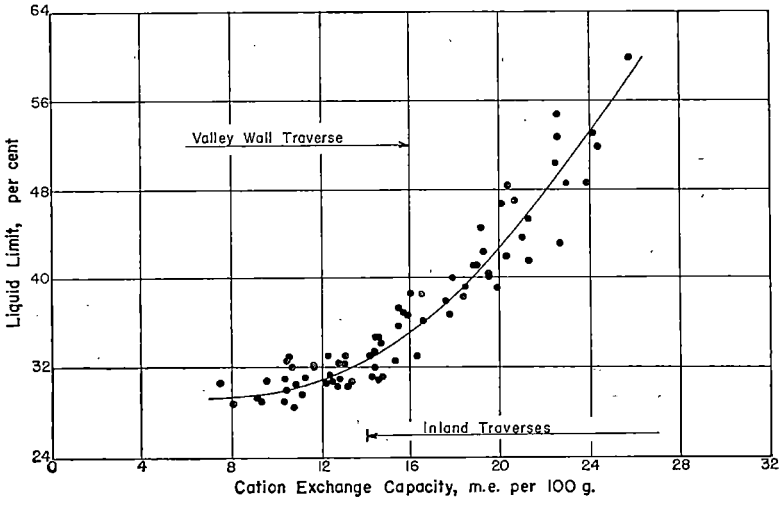

Fig. 8. Relation of cation exchange capacity to liquid limit.

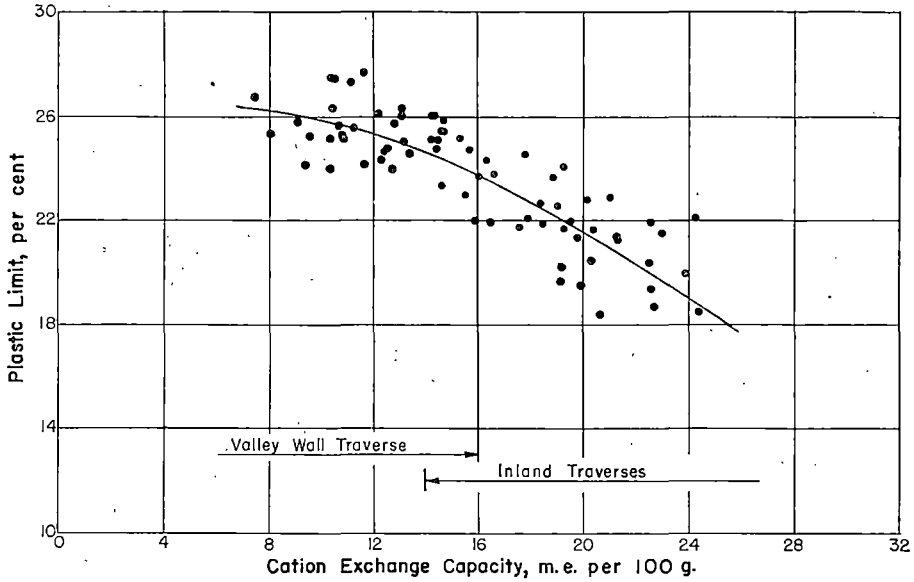

Fig. 9. Relation of cation exchange capacity to plastic limit.

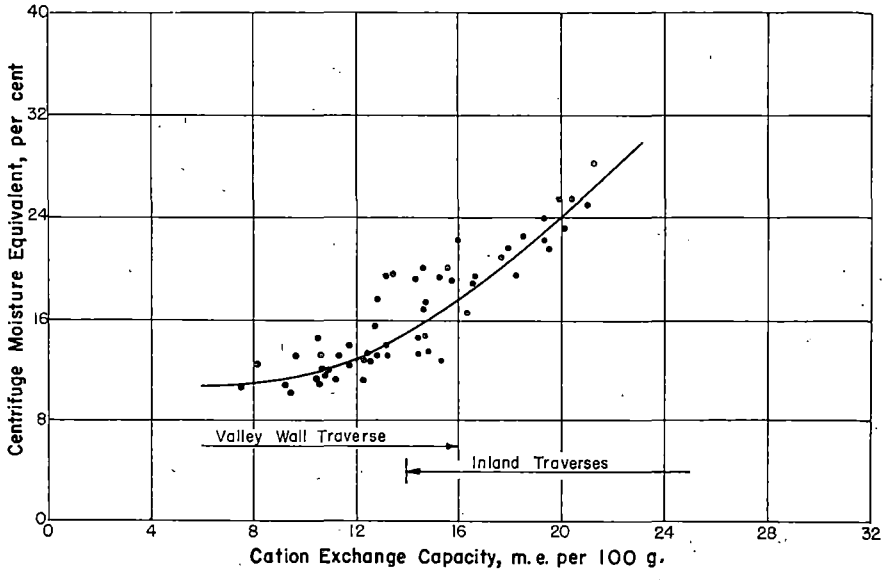

Fig. 10. Relation of cation exchange capacity to plasticity index.

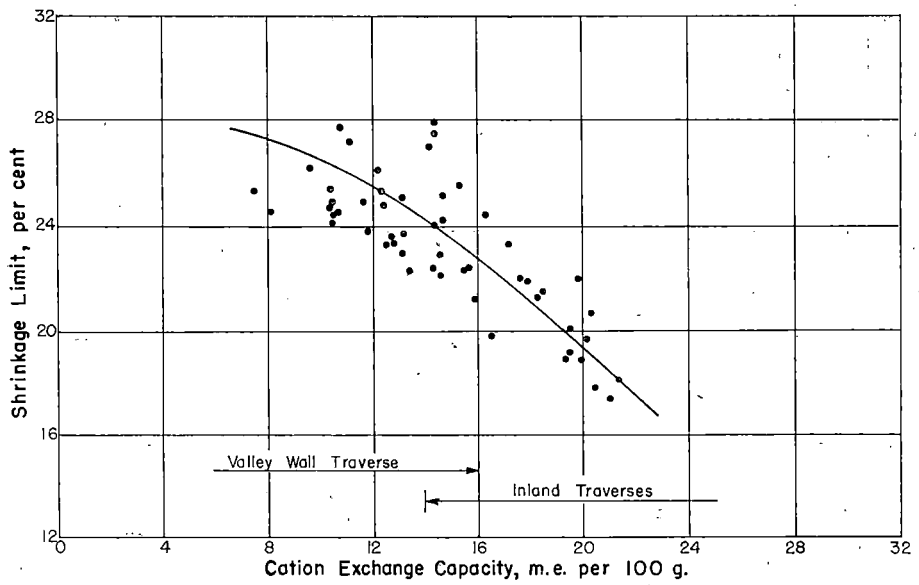

Fig. 11. Relation of cation exchange capacity to shrinkage limit.

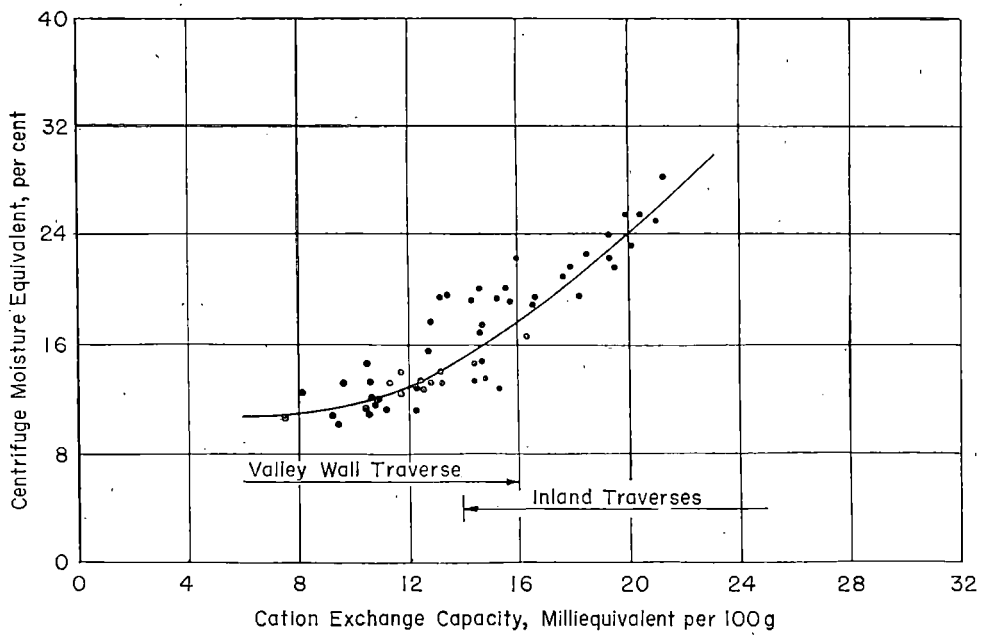

Fig. 12. Relation of cation exchange capacity to centrifuge moisture equivalent.

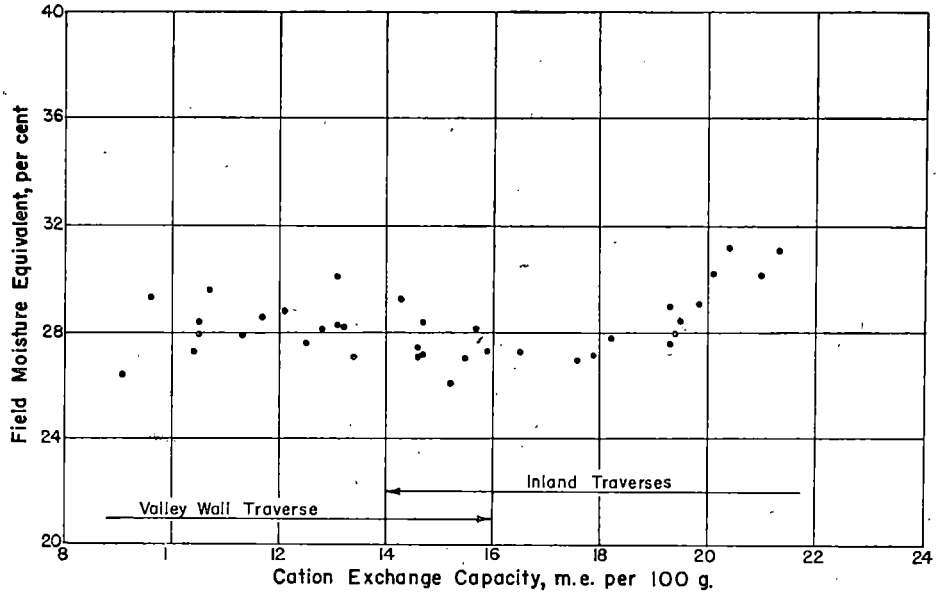

Fig. 13. Relation of cation exchange capacity to field moisture equivalent.

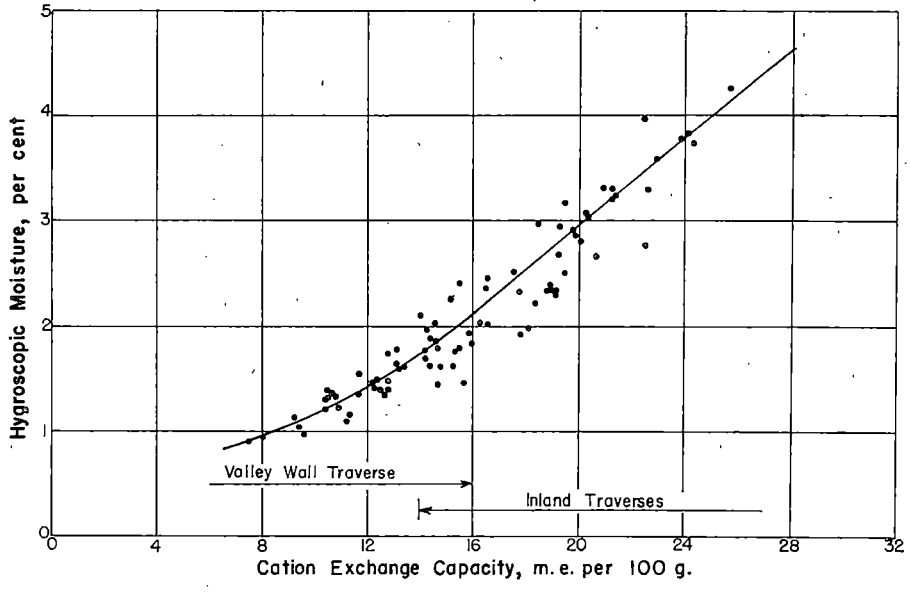

Fig. 14. Relation of cation exchange capacity to hygroscopic moisture.

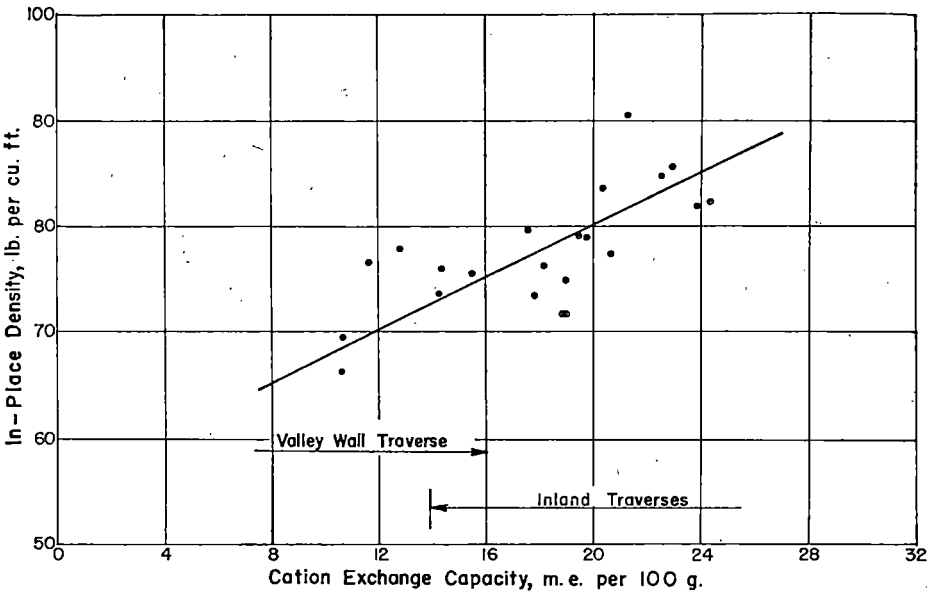

Fig. 15. Relation of cation exchange capacity to in-place density.

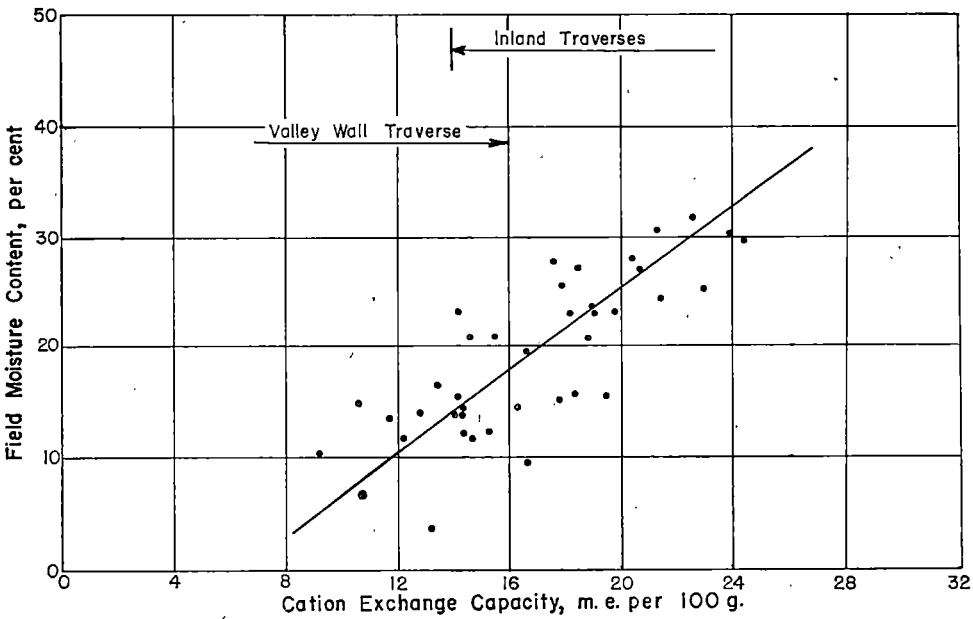

Fig. 16. Relation of cation exchange capacity to field moisture content.

The results of the correlation study show that the following engineering properties of Wisconsin loess have a curvilinear relationship with cation exchange capacity:

1. Amount of 0.002 mm. clay (figure 7)
2. Liquid limit (figure 8)
3. Plastic limit (figure 9)
4. Plasticity index (figure 10)
5. Shrinkage limit (figure 11)
6. Centrifuge moisture equivalent (figure 12)
7. Hygroscopic moisture (figure 14)

Reference to the curves shows that the amount of 0.002 mm. clay, the liquid limit, the plasticity index, the centrifuge moisture equivalent, and the hygroscopic moisture increase in value with an increase in cation exchange capacity, while the plastic limit and the shrinkage limit decrease in value with an increase in cation exchange capacity. For the most part, the slopes of the curves change gradually with an increase of cation exchange capacity up to about fourteen to sixteen milliequivalents per 100 g, which, as marked on the graphs, is the cation exchange capacity range common to both east valley wall samples and samples from the four inland traverses. Above this range, where the plotted points represent samples of variable clay content, but of low and practically uniform carbonate content, the curves become steeper and approach a straight line. This suggests that the curvilinear relationships may be due at least in part to the variation of carbonate content in the loess.

A limited amount of experimental investigation indicates that the presence of carbonates tends to lower the cation exchange capacity of the "whole" loess. For this reason, the high carbonate content valley wall traverse samples had their cation exchange capacities reduced more than the low carbonate content samples obtained along the inland traverses. The amount of 0.002 mm. clay has been correlated with engineering properties of the same Wisconsin loess samples[6]. The engineering properties which show a curvilinear relationship to cation exchange capacity in the present study showed a linear relationship to amount of 0.002 mm. clay. This difference indicates that cation exchange capacity may be more sensitive to the variation of carbonate content in the samples than are the engineering properties.

The field moisture equivalent shows no well defined relationship with cation exchange capacity. The data, however, does indicate that it varies slowly as cation exchange capacity increases (figure 13). Correlations of

the field moisture equivalent and clay content in previous investigations have shown that the F.M.E. varies slowly as clay content increases and even for a given type of soil exhibits considerable variability[6, 13]. More plotted points in the cation exchange capacity range above 16 m.e. per 100 g are needed before a significant curve can be fitted; for this reason no curve is shown.

On the basis of the data plotted both the in-place density, providing in-place density measurements are made in similar stratigraphic positions, and the field moisture content of Wisconsin loess appear to be directly proportional to cation exchange capacity, showing an increase in value with an increase in cation exchange capacity (figure 15, 16).

CONCLUSIONS

1. The quantitative measurement of the amount of ammonium (NH_4+) adsorbed upon leaching or shaking the sample thoroughly with neutral normal ammonium acetate salt solution is a relatively simple and accurate method for determining the cation exchange capacity of Wisconsin loess.
2. The data available at the present time indicate that the major variables in the Wisconsin loess are the amounts of clay and carbonates present.
3. The variation of cation exchange capacity in Wisconsin loess is attributed mainly to the variation in the amount of clay present in the loess. Studies to date indicate that the clay mineral composition is uniform and that minerals of the illite group predominate.
4. The effect of carbonate content on cation exchange capacity and on engineering properties needs additional study.
5. Since the determination of cation exchange capacity is comparatively sensitive to the test technique employed, a standard test method for determining it should be adopted to place the results of different investigators on a comparable basis.
6. On the basis of tests on over one hundred samples of Wisconsin loess, the range in cation exchange capacity values is from 7.5 to 25.8 m.e. per 100 g of oven dry loess.
7. Cation exchange capacity is quantitative and significant in character and has direct application to engineering soil problems. By means of correlations such as those presented, cation exchange capacity can be directly related to conventional engineering properties of soils.

APPENDIX

TEST METHODS USED FOR MEASURING CATION EXCHANGE CAPACITY

The test methods used in measuring the cation exchange capacity of the Wisconsin loess samples are presented in the following order: exchange

of cations, distillation, titration, and calculations.

Exchange of Cations

The exchange of cations was by two methods. The first method, which was used with about half of the loess samples, was to leach the sample in a carbon filter tube funnel with neutral normal ammonium acetate solution. The pH of the normal solution may be adjusted to pH 7.0 by the use of either ammonium hydroxide or acetic acid, depending on the initial pH. In the second method, the exchange was accomplished by shaking a mixture of soil and ammonium acetate solution in a bottle. Instructions for using the leaching method are presented first.

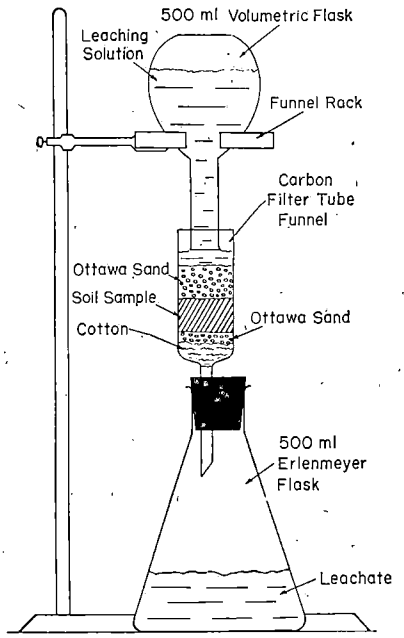

Fig. 17. Leaching apparatus for cation exchange capacity determination.

The leaching apparatus is shown in figure 17. A pad of cotton is placed in the bottom of the carbon filter tube funnel and covered with 0.25 in. of Ottawa sand which has no effect on the cation exchange capacity. Approximately ten grams of air dry loess, ground to pass the No. 40 sieve (100 percent passed), is accurately weighed to the nearest 0.001 g and placed loosely on top of the sand layer in the carbon filter. The hygroscopic moisture in the air dry material should be determined on a duplicate sample of

loess in accordance with the procedure given in ASTM Method D 422-51. Another 0.25 in. layer of sand is then placed over the loess sample to prevent roiling when the leaching solution is brought into contact with the sample. A volumetric flask containing 250 ml. of neutral normal ammonium acetate solution is inverted over the top of the sand layer (figure 17), and the leaching rate is adjusted to not more than fifteen drops per minute, since a more rapid rate appears to give cation exchange capacity results which are too low. The leaching rate can be slowed by pressing down on the sand layer with the mouth of the flask. When the ammonium acetate solution leaching is completed, the sample is leached again with 150 ml. of neutral 70 percent methyl alcohol solution to wash out any excess ammonium acetate solution that may be held in void spaces. The next step is to determine quantitatively the amount of ammonium ions held by the sample.

Shaking the loess sample with neutral normal ammonium acetate solution will also bring about the desired exchange cations. This second method, which was used in the latter part of the loess study, requires the use of a centrifuge. The weighed air dry loess sample is placed in a rubber stoppered centrifuge bottle and shaken, by hand or electric shaker, with 250 ml. of ammonium acetate solution for three minutes. The suspension is then centrifuged until all soil particles are packed in the bottom of the bottle. The clear supernatant liquid above the sample is suction filtered through a Buechner funnel fitted with a dense filter paper, and the centrifuge bottle is refilled with 250 ml. of fresh ammonium acetate solution. After the shaking and centrifuging operation has been repeated, the supernatant liquid is filtered through the Buechner funnel, and the soil is washed into the funnel and filtered. (Experiments with Wisconsin loess samples indicated no significant increase in cation exchange capacity when the shaking and centrifuging operation was repeated a third time.) The soil cake is then washed with 150 ml. of neutral 70 percent methyl alcohol solution before determining the amount of ammonium ions adsorbed by the soil.

Both methods of determining exchange appear to give about the same degree of cation exchange with loess samples. However, the shaking method is less time consuming, and for this reason it is more satisfactory for engineering laboratory use.

Distillation

Immediately following the alcohol washing the loess sample containing ammonium that has been taken up by exchange is transferred to a 500 ml. Kjeldahl flask and covered with 150 ml. of distilled water (figure 18). If the leaching method has been used, the entire contents of the carbon filter tube funnel are washed into the Kjeldahl flask. The transfer of soil from the Buechner funnel used in the shaking method is best accomplished by rolling up the loess sample in the filter paper and transferring paper and

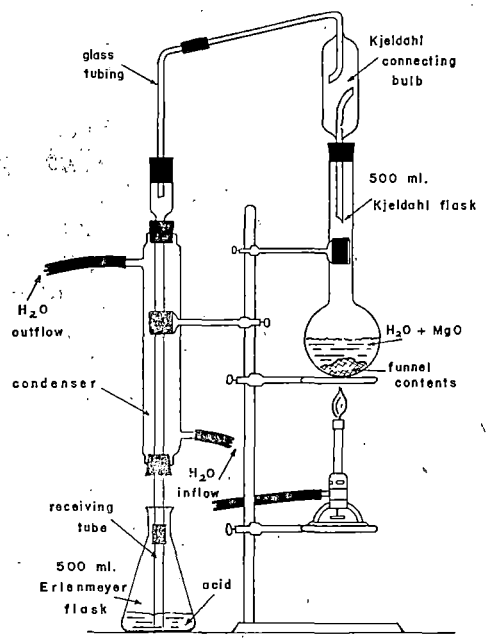

Fig. 18. Distillation apparatus for cation exchange capacity determination.

sample to the Kjeldahl flask. Wetting the caked soil slightly facilitates this operation. Soil grain clinging to the sides of the funnel may be transferred by wetting a clean sheet of filter paper with distilled water and wiping the inside of the funnel clean; the filter paper is then placed in the flask.

Next exactly 50 ml. of 0.1000 N hydrochloric acid should be measured and placed in a 500 ml. Erlenmeyer flask. The flask should be placed under the condenser with the receiving tube of the condenser extending well into the acid (figures 18, 19). One ml. of 0.2 percent methyl red solution should be added to the acid and a full teaspoon of magnesium oxide to the contents of the Kjeldahl flask. All connections of the distillation apparatus should be checked to make sure they are tight enough to prevent any escape of ammonia. Then the burner should be lighted under the Kjeldahl flask and the contents distilled nearly to dryness.

Care should be exercised throughout the distillation to prevent acid from being sucked up into the Kjeldahl flask. Should the acid start to be sucked up into the condenser, the connecting line at the top of the condenser should be momentarily opened. Care should be taken that the flame is on throughout the distillation, as a loss of heat source will create a vacuum in the distillation system and suck acid up into the condenser. At the end

of the distillation, the Kjeldahl connecting bulb should be disconnected before removing the flame.

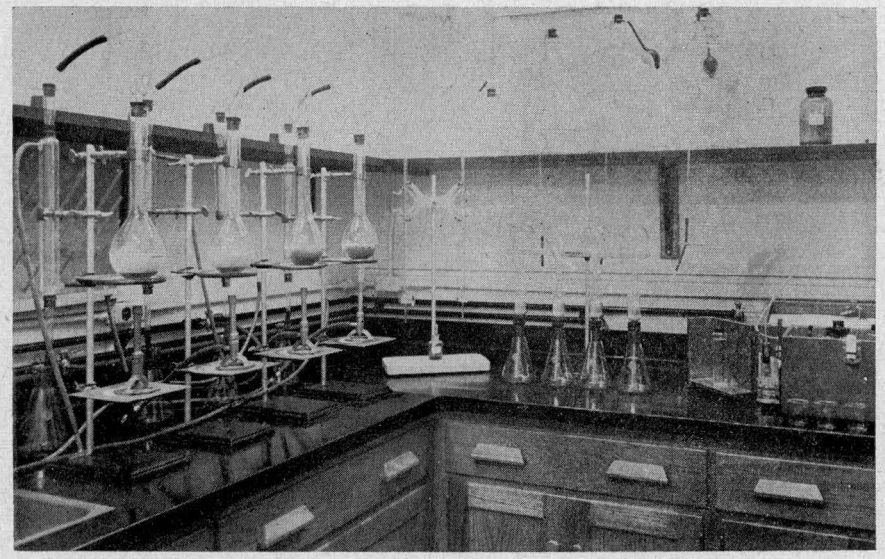

Fig. 19. Arrangement of cation exchange capacity apparatus for testing four samples at the same time. Left to right: Distillation apparatus, titration apparatus, leaching apparatus, and pH meter. The bottles on the shelf above contain chemicals used in the tests.

Titration

The Erlenmeyer flask should be removed and the acid washed off the receiving tube into the flask with distilled water. The excess acid should be titrated with 0.1000 normal sodium hydroxide, reading the burette to the nearest 0.1 ml. The end-point of the titration occurs at a pH of exactly 6.2. The end-point can be determined most accurately by the use of a pH meter, though the end-point is evidenced by a yellow orange color. At a pH of about 5.7 the color turns from red to red orange and then fades to yellow orange as the titration proceeds to pH 6.2; there is a difference of 0.5 to 0.7 ml. between pH 5.7 and 6.2.

Calculations

The cation exchange capacity of the loess sample may now be calculated in milliequivalents per 100 g of oven dry soil.

The oven dry weight of the sample used is determined by correcting the air dry weight for hygroscopic moisture as follows:

$$d = a\frac{100}{100 + p} \quad (1)$$

where:

d = wt. of oven dry sample in grams
a = wt. of air dry sample in grams
p = percent hygroscopic moisture

The cation exchange capacity is calculated from the following equation:

$$c = \frac{A - B}{d} 100 \quad (2)$$

where:

c = cation exchange capacity in m.e. per 100 g
A = (ml. of HCl used) (normality of HCl)
B = (ml. of NaOH used) (normality of NaOH)
d = wt. of oven dry sample in grams

In general, cation exchange capacity values should be reported as the average of at least two determinations.

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CATION EXCHANGE CAPACITY OF THE CLAY FRACTION OF LOESS IN SOUTHWESTERN IOWA

by

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J. B. Sheeler, Associate Professor, Civil Engineering

(Iowa Academy of Science Proceedings, 60:354-361. 1953.)

The cation exchange capacity of clay size material extracted from soil with a low organic matter content depends on the kinds of clay minerals in the soil. If most of the extracted clay is of one kind of clay mineral, the exchange capacity will indicate what that mineral is. Cation exchange capacity determinations were made on the minus 2 micron clay size range of selected samples of loess from the southwestern Iowa area (figure 1).

WHOLE LOESS SAMPLES

The origin, distribution, and property variations of the Wisconsin loess, also referred to as Peorian loess, which mantles much of the southwestern Iowa area (figure 1) have been discussed[2, 3, 4]. The minus 2 micron clay fractions used in this study were extracted from eleven samples of whole loess which were selected as representing the range in properties of the more than 150 loess samples that have been tested in the property variation studies of the Iowa Engineering Experiment Station.

The locations from which the whole loess samples were taken are given in table I, and the sampling locations are shown in figure 1. Sample 55-1 is Upper Wisconsin or Cary-Mankato loess from the Pisgah road section[10]. The other Wisconsin age loess samples are undifferentiated because the buried (Brady) soil[11], which separates the upper and lower Wisconsin components, was not present. The samples of Loveland soil, Sangamon soil profile on Loveland loess, and of Loveland loess from the type section at Loveland, Pottawattamie County, were included in the study for comparative purposes.

Table II gives some properties of the whole loess which are indicative of the cation exchange material in the samples. The Wisconsin loess samples are arranged in the order of increasing clay content. The range in clay content of the Wisconsin loess in southwestern Iowa is shown. Whether the type section of Loveland samples used are representative of Loveland soil and loess exposed elsewhere in southwestern Iowa is not as yet known. The Loveland soil had a higher clay content than the underlying loess, and both

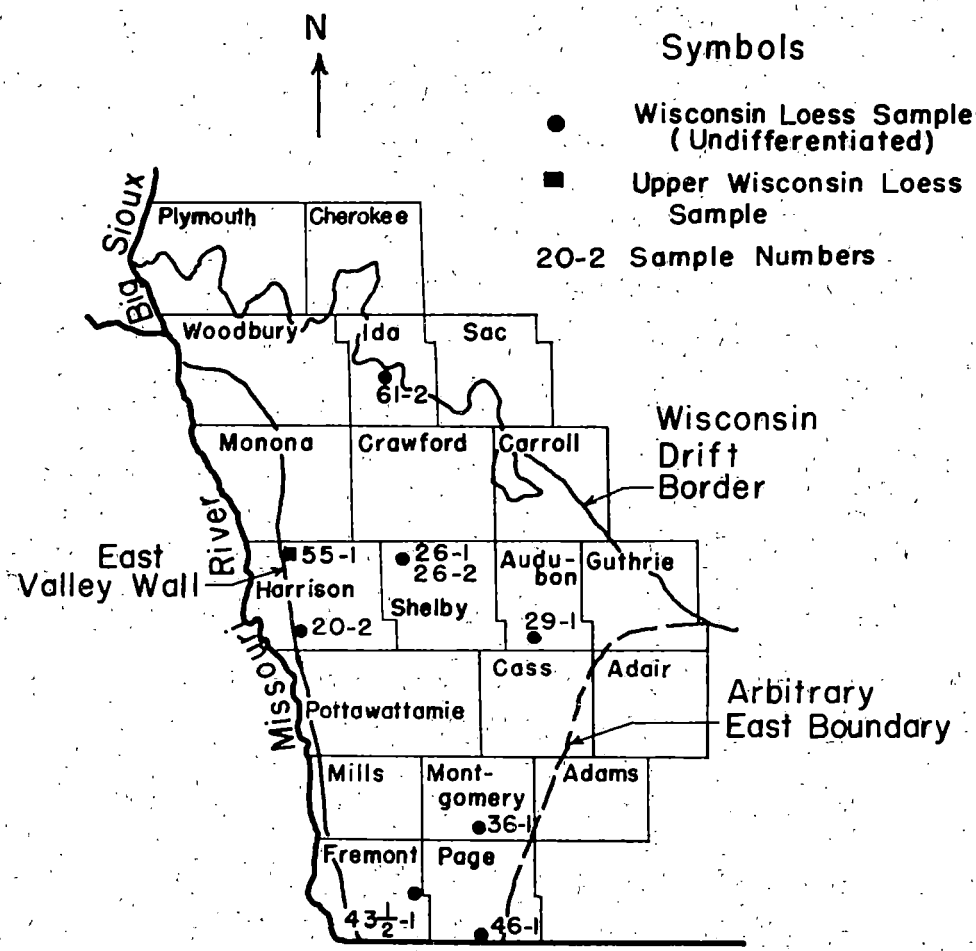

Fig. 1. Distribution of sampling locations in southwestern Iowa loess area. Symbols indicate age classification of loess sampled.

Loveland samples had lower clay contents than some of the more plastic Wisconsin loess samples.

Sample 26-2 is from the same location as sample 26-1 but was taken at a greater depth in the Wisconsin loess section. Both samples contain practically the same amount of clay. However sample 26-2 is unoxidized and unleached, and sample 26-1 is oxidized and leached. Sample 26-2 was included to determine what effect oxidation and leaching might have on the clay mineral in the loess. This was the only unoxidized sample used in the study.

The organic matter content of all samples was low, and for this reason the inorganic clay minerals are considered to be largely responsible for cation exchange. Carbonate contents were variable, being relatively high for

unleached samples taken near the major source areas of the loess[2]. The distribution of carbonates in the different particle size fractions of the loess has not been determined. The pH values varied from 6.7 to 8.7 or from near neutral to alkaline. The variations in whole loess cation exchange capacity,

TABLE I. LOESS SAMPLING LOCATIONS IN SOUTHWESTERN IOWA

Sample No.	Material	Age classification	Sampling depth* (ft.)	County	Location			Soil Series
					Section	Township North	Range West	
55-1	Loess	Upper Wisconsin†	2½—3½	Harrison	SW/c,S-8	81	44	Hamburg
20-2	"	Wisconsin (Undifferentiated)	39—40	Harrison	S-15‡	78	43	Hamburg
61-2	"	"	17—18	Ida	NW¼,S-9	87	40	Monona
26-1	"	"	4—5	Shelby	SE¼,S-21	81	40	Monona
26-2	"	"	10—11	"	"	"	"	"
29-1	"	"	5—6	Audubon	NW/c,SW¼,S-13	78	36	Marshall
36-1	"	"	5½—6½	Montgomery	SE¼,NE¼,S-14	72	38	Marshall
43½-1	"	"	5—6	Fremont	NW/c,S-36	68	40	Marshall
46-1	"	"	5—6	Page	NW¼,S-30	67	37	Marshall
49-3	Soil§	Loveland	55**	Pottawattamie	SE/c,NW¼,S-3††	77	44	Hamburg
49.4	Loess	"	65‡‡	"	"	"	"	"

* Measurements are from earth's surface.
† Also called Cary-Mankato loess and Bignell loess. Sampled from Pisgah road section (4).
‡ Sampled from bluff behind third ward school in city of Missouri Valley.
§ Sangamon soil profile on Loveland loess.
**Sampled from about middle of 10 ± ft. Sangamon soil profile.
††Sampled from type section of Loveland loess at northeast edge of town of Loveland (5).
‡‡Sampled in Loveland loess about 3 ft. above slump.

TABLE II. SOME PROPERTIES OF WHOLE LOESS SAMPLES

Sample No.	Age Classification	Textural Composition.. Sand (%)	Silt (%)	Clay (%) —5μ	—2μ	Organic matter (%)	Carbonates (%CaCO3)	Oxidation	pH	Cat. Ex. Cap (m.e./100g)	Plasticity Index (%)	B.P.R.† Classification
55-1	Upper Wisconsin Wisconsin	4.0	82.6	13.4	12.0	0.24	11.0	Oxidized	8.4	11.2	2.3	A-4(8)
20-2	(Undifferentiated)	1.4	78.8	19.8	16.0	0.17	10.2	Oxidized	8.7	13.4	6.2	A-4(8)
61-2	"	5.2	70.8	24.0	19.8	0.15	11.5	Oxidized	8.3	14.2	10.8	A-6(8)
26-1	"	2.0	70.6	27.4	22.4	0.18	1.4	Oxidized	7.0	18.2	12.5	A-6(9)
26-2	"	0.9	69.8	29.3	23.1	0.17	8.7	Unoxidized	8.3	17.9	17.8	A-6(9)
29-1	"	1.0	67.9	31.1	25.0	0.25	2.9	Oxidized	8.3	19.5	18.0	A-6(11)
36-1	"	0.8	63.2	36.0	28.9	0.21	1.8	Oxidized	6.7	21.0	20.7	A-7-6(13)
43½-1	"	0.4	60.2	39.4	33.0	0.37	0.5	Oxidized	6.7	24.4	33.4	A-7-6(18)
46-1	"	0.8	55.7	43.5	36.2	0.30	1.5	Oxidized	6.3	22.6	32.7	A-7-6(19)
49-3	Loveland (soil)	2.7	61.1	36.2	31.4	0.19	1.3	Oxidized	8.2	22.7	24.6	A-7-6(15)
49-4	Loveland	5.3	65.9	28.8	24.6	0.11	7.0	Oxidized	8.2	16.6	15.3	A-6(10)

*Sand—2.0 to 0.05 mm., Silt—0.05 to 0.005 mm. One micron equals 0.001 mm.
†Bureau of Public Roads Soil Classification System. Also referred to as the Highway Research Board System or the American Association of State Highway Officials (AASHO) System[1].

in plasticity index, and in BPR classification are mainly due to the variation in clay content[4].

CLAY FRACTION

The minus 2 micron portions of the whole loess samples were used for the exchange capacity determinations because practically all of the cation exchange material, the clay minerals, occur in this particle size range. Complete separation of the clay minerals from other substances such as quartz and carbonates is difficult, but only very small amounts of such substances are commonly found in the minus 2 micron soil fraction.

The separation of the minus 2 micron clay material from the whole loess was by means of a sedimentation procedure. In this procedure neither the whole loess nor the separated clay was given hydrochloric acid or hydrogen peroxide treatments. When a deflocculating agent was needed to prevent flocculation, 0.1 N sodium hydroxide was used.

DETERMINATION OF CATION EXCHANGE CAPACITY

Cation exchange capacity can be determined by a number of methods, most of which involve leaching the soil sample with a salt solution containing known cations followed by analysis either of the resulting soil or of the solution for the amount of cations exchanged[9].

The cation exchange capacity of a soil denotes the total amount of cations that can be exchanged under a given set of conditions and not necessarily the amount that could be exchanged under other conditions. The determination is particularly sensitive to the pH of the salt solution; the common practice is to use a neutral (pH = 7) solution. Neutral normal ammonium acetate has been found to be a salt solution especially well adapted to the exchange capacity determination[9]. With this solution the exchange capacity of inorganic soils can be determined with reasonable accuracy even when the soil contains soluble salts and calcium carbonate.

Test methods used in determining cation exchange capacities of whole Wisconsin loess samples have been previously presented[4]. A step by step summary of the procedure used in the present study is as follows:

1. Weigh out about one gram (accurate to 1 mg) of representative air dry clay and place in a 300 ml. centrifuge bottle. (A similar sample should be weighed out for the hygroscopic moisture determination needed to convert air dry weight to oven dry weight.)

2. Add 10 g of fine Ottawa sand to the clay in the centrifuge bottle. (Ottawa sand is inert and increases the permeability for the purpose of filtration in Step 7.)

3. Add 250 ml. of neutral normal ammonium acetate to the contents of the centrifuge bottle and shake for three minutes. (Higher normalities and increased shaking times were experimented with but did not significantly affect results.)

4. Centrifuge at 2000 RPM for ten minutes.

5. Decant the clear supernatant liquid.

6. Repeat step 3.

7. Filter the contents of the centrifuge bottle with a Buechner funnel containing two fine filter papers.

8. Wash the material retained on the filter paper with 150 ml. of neutral 70 percent (by volume) methyl alcohol to remove the excess ammonium acetate trapped in void spaces.

9. Determine the amount of ammonia held in the exchange positions of the clay by a modified Kjeldahl nitrogen determination[4].

10. Calculate the cation exchange capacity in milliequivalents per 100 g of oven dry clay[4].

CATION EXCHANGE CAPACITIES

Cation exchange capacities of minus 2 micron clay fractions are given in table III. Since the clay minerals are the primary seat of cation exchange, the uniformity of the data indicates that in the kinds of clay minerals in the Wisconsin loess the exchange capacity varies little. The data further indicate that there is little difference between the clay fractions of the Wisconsin loess and the Loveland loess. The results of differential termal analyses on whole loess samples substantiate this[2].

TABLE III. CATION EXCHANGE CAPACITIES OF MINUS 2 MICRON CLAY FRACTIONS

Sample No.	Material	Age Classification	Cation Exchange Capacity* (m.e./100g)
55-1	Loess	Upper Wisconsin	59.3
20-2	"	Wisconsin (Undifferentiated)	58.6
61-2	"	"	52.5
26-1	"	"	62.8
26-2	"	"	59.0
29-1	"	"	62.2
36-1	"	"	63.1
43½-1	"	"	59.9
46-1	"	"	57.4
49-3	Soil	Loveland	63.4
49-4	Loess	"	54.4

*Values reported are the average of two determinations.

Slight variations in the cation exchange capacity values in table III may or may not be significant. Further studies are in progress to determine whether they are due to experimental factors or to slight variations in mineral composition.

The clay fractions extracted from soils are rarely composed of a single kind of clay mineral but usually contain two or more mixed with other substances from which complete separation is difficult. The exchange capacity of the minus 2 micron particle size range for this reason can at best be used only to estimate roughly the predominant kind of clay mineral present. The estimation can be made by comparing the determined value with the exchange capacities of comparatively pure clay minerals. Cation exchange capacities of the common clay minerals are given[5] as:

Montmorillonite	60-100 m.e./100g
Attapulgite	25-30
Illite	20-40
Kaolinite	3-15
Halloysite	6-10

A comparison of the data in table III with these values indicates that the loess clay fractions contain a predominance of montmorillonite group minerals. Differential thermal curves for samples of whole loess from southwestern Iowa have indicated the presence of illite[2]. This mineral is often associated with montmorillonite in soils, and its presence would tend to lower the exchange capacity[8].

Montmorillonite can develop from illite by weathering[6], which may be why the Loveland soil has a higher exchange capacity than the loess on which it developed. The same explanation may apply to the difference in the exchange capacities of samples 26-1 and 26-2. The oxidized and leached samples (26-1) had a slightly higher exchange capacity than the unoxidized and unleached sample (26-2).

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X-RAY FLUORESCENCE ANALYSIS OF TOTAL IRON AND MANGANESE IN SOILS

by

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(Iowa Academy of Science Proceedings, 66:283-301. 1959.)

ABSTRACT

X-ray fluorescence analysis, also known as X-ray spectroscopy, is a rapid, accurate and non-destructive means of qualitative and quantitative chemical analysis which requires relatively little sample material. Irradiation of a sample by hard, short wavelength X-rays causes the elements within the sample to emit secondary X-rays which are characteristic of and specific for the various elements present. The intensities of such secondary X-rays are proportional to concentrations of the emitting elements, and linear relationships may be shown at low or high concentrations of the element. Two methods are presented for the quantitative determination of total iron and manganese in soils. The first necessitates the preparation of a calibration curve and is applicable only to soils having similar properties. In this method known increments of Fe_2O_3 and/or MnO_2 are added to the original soil and X-ray intensities are obtained. Analysis of an unknown then utilizes the resulting equation which expresses the relation between intensity and concentration.

A second method, here called the two-point method, is described and evaluated. This method does not require a calibration curve and is valid for a wider range of samples. A sample is analyzed for the element, either iron or manganese, and intensities are obtained, then a known amount of the element is added to the sample and intensities are again measured. A suitable equation utilizing the two intensities for the determination of the element in the original sample is presented. Data are included to indicate that this method is satisfactory and accurate.

Materials subjected either to a high voltage electron beam or to a high energy X-ray beam absorb part of the beam energy and re-emit it as X-rays. The X-ray spectrum thus generated is characteristic of the elements in the sample, so the measurement of the emitted wavelengths is a means for

spectrochemical analysis. Since emission intensities are proportional to concentrations of emitted elements in the sample, quantitative relationships may also be set up.

In the commercial X-ray spectroscope the sample is irradiated with hard, short wavelength X rays from a tungsten tube. Secondary radiation emitted by the sample is collimated, then it is analyzed by diffraction from a known crystal (figure 1) which in effect separates the beam into component wavelengths. The θ angle at which diffraction will occur appears in the Bragg equation

$$n\lambda = 2d \sin \theta$$

where n = a whole number, indicating the order of diffraction
λ = X-ray wavelength
d = interplanar spacing in the crystal
θ = the angle between the X-ray beam and the reflecting planes in the crystal.

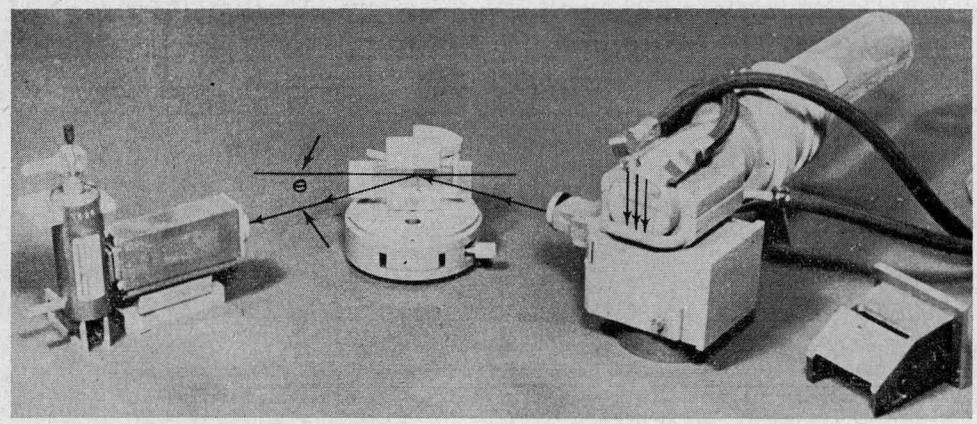

Fig. 1. Schematic arrangement for X-ray fluorescence analysis. Hard X-rays from a tungsten tube shoot down (triple arrow) and irradiate the sample contained in a sample drawer. Fluorescent X-rays from the sample are then diffracted by a known crystal and detected with a counter tube, shown at right. The θ angle is specific for diffraction of a given wavelength.

In X-ray spectroscopy d is known and θ is measured, then the equation is solved for λ. This is in contrast with X-ray diffraction analysis, where λ is known and θ is measured, and the equation is solved for d.

For qualitative identification of elements in the sample, the θ angle may be scanned while diffracted intensities are continuously measured with a Geiger-Muller or scintillation counter and the counting rate recorded on a strip chart recorder. Each peak intensity then indicates a θ angle at which the Bragg equation is satisfied, and the emitted element may be identified

from its wavelength by means of prepared tables. The identification may be checked at the various corresponding θ angles.

For quantitative measurement, the θ angle is set for the desired wavelength, and counts are made for measured time intervals. The X-ray intensity is then expressed in counts per second and is corrected for instrumental drift or crystal misalignment by re-run of a standard. A correction is also made for scattered radiation or background; this is made by subtracting counts at a nearby θ angle which does not coincide with the θ for other wavelengths emitted by other elements in the sample.

Unless objectionable overlapping of wavelengths from different elements occurs, the radiation usually selected for measurement is the strongest radiation, the Kα. Tungsten radiation applied to the sample causes ejection of electrons from inner shells to outer shells of the atoms; when the electrons fall back, the change in energy level results in fluorescent emission of X-rays. Kα results from vacancies in the innermost K shell being filled by electrons from the next outer shell, the L shell. Radiation designated Kβ is emitted when vacancies in K shell are filled from the third, or M shell. Since this involves a greater change in energy level, Kβ has a higher energy, i.e., a shorter wavelength. However, since filling of the K from the neighboring L is statistically more probable, Kα radiation is the more intense.

ABSORPTION

Ideally a fluorescent intensity is proportional to the mass concentration of emitted atoms in the surface of the sample. At low or high concentrations of an element this is approximately true, and a linear relationship between intensity and concentration may be found.

TABLE I. MASS ABSORPTION COEFFICIENTS OF DIFFERENT ELEMENTS FOR FE Kα AND MNKα RADIATIONS

Element	Atomic Number	μ/ρ FeKα* (1.9373 A)	MnKα† (2.103 A)
Mg	12	75.7	
Al	13	92.8	115
Si	14	116.3	
Ca	20	317	380
Cr	24	490	
Mn	25	63.6	
Fe	26	72.8	90

*Internationale Tabellen zur Bestimmung von Kristallstrukturen, Vol. 2, 1935.

†Interpolated from values in Handbook of Chemistry and Physics, Chemical Rubber Publishing Co., 35th Ed., Cleveland, Ohio, 1953.

With intermediate concentrations the linear relationships no longer hold, illustrating the effect of re-absorption of emitted rays before they leave the sample. The amount of absorption varies widely depending on the X-ray wavelength and the identity of the elements in the sample. Mass absorption coefficients of different common soil elements for iron $K\alpha$ and manganese $K\alpha$ radiations are shown in Table 1. As an example, the presence of calcium in a sample will reduce the fluorescent iron $K\alpha$ intensity far more than the presence of an equal amount of magnesium. Similarly, at high iron content the low absorption factor of iron for iron $K\alpha$ radiation ordinarily will mean disproportionately high iron $K\alpha$ intensities. Fluorescent intensities are also affected by absorption of tungsten radiation; however these absorption coefficients are so low they have relatively little effect.

Several solutions have been suggested for the absorption problem. The simplest is to prepare a calibration curve specific for soils having similar physical and chemical properties, and assume that absorption does not appreciably vary from sample to sample. Such a curve is presented for determination of manganese in a friable calcareous loess. Measured increments of MnO_2 were added to samples of the loess and the fluorescent intensities were measured; an equation relating intensity to MnO_2 content was then found by the method of least squares.

Another method is to add a measured amount of a reference standard to a sample and calibrate on the basis of emission intensity ratios between the unknown and the standard. The standard contains an element whose radiation is absorbed about the same as radiation from the element to be measured, which means that it will emit nearly the same wavelength and will have nearly the same atomic number. For example, for measurement of iron, a carefully measured amount of a cobalt compound might be added to the sample, since the atomic number of cobalt is one higher than that of iron. In general the absorption of cobalt $K\alpha$ by different elements is much the same as and about proportional to the absorption of iron $K\alpha$. However, notable exceptions occur for elements near the absorption edge, which ordinarily occurs about two atomic numbers below the emitting element. That is, cobalt $K\alpha$ (at. no. 27) is greatly absorbed by manganese (at. no. 25). Similarly iron $K\alpha$ (at. no. 26) is most strongly absorbed by chromium (at. no. 24). Therefore presence of variable amounts of manganese in a soil would strongly influence iron $K\alpha$ to cobalt $K\alpha$ intensity ratios and would affect the measurement of iron.

Another possibility for use of an internal standard is to go the other way in atomic number and select the next number lower. For measurement of iron, this would mean the use of manganese. Unfortunately, since soils already contain manganese, the intensities from the internal standard would be increased. Other standards may be selected, but the greater the difference in emitted wavelength between the standard and iron, the greater the difference in absorption factors.

Because these solutions are not entirely satisfactory, a different approach was tried. A measured quantity of the same material is added, in this example a compound of iron, and the original iron content is determined by algebraic extrapolation, here called the two-point method. So far the method has been used only in the linear response range (i.e. less than about 10% Fe_2O_3), but it should have good accuracy because of the identical absorption factors.

PROCEDURE

Standard samples were prepared with a friable, calcareous loess (Sample No. 20-2 VII) known to be low in manganese and iron. Carefully weighed increments of Fe_2O_3 or MnO_2 were added to the previously air dried and pulverized soil in 1 oz. wide-mouth glass bottles; glass beads were added as a mixing aid, and the bottles were put on a rotary mixer for a minimum of one hour.

Samples were packed dry into 1″ x 1″ x $\frac{1}{10}$″ bakelite holders and subjected to 50 kvp tungsten radiation. The tube was operated at 50 ma. A sodium chloride analyzing crystal was used, and the diffracted radiation was counted with a proportional counter tube. The count rates reported are from averages of ten 10 second counts. A General Electric XRD-5 unit was used for the analysis.

RESULTS

Manganese

Calibration data from the prepared MnO_2 reference samples are in Table II. Conversion of percents added to total percents requires some algebraic gymnastics, since the base for calculation of percent changes. That is, the added amounts are expressed as percentages of the total mix, but the constant amount originally in the soil is not a percent of total mix since the amount of the total mix changes as more material is added.

TABLE II. MANGANESE $K\alpha$ CALIBRATION DATA FOR FRIABLE LOESS SOIL

% added MnO_2	Total % MnO_2*	MnK_α, cps† ($2\theta = 43.79°$)
0	0.1536	57.8
0.05	0.204	77.1
0.10	0.253	102.0
0.15	0.303	110.7
0.20	0.353	135.0
0.25	0.403	150.6
0.30	0.453	169.3

*0% value from equation 4; all others from column 1 and equation 1.
†Corrected for background of 14.7 cps ($2\theta = 47.0°$), and to reference count of 28,200 cps on pure powdered MnO_2.

If C is the added concentration of MnO_2 (for convenience expressed as a

fraction), if X_0 is the concentration in the original sample, and if X is the total concentration, X at any point equals C plus X_0 corrected for the change in base, or

$$X = C + X_0 (1 - C). \tag{1}$$

X_0 may be evaluated from the plot of intensity vs. added concentration, which will take the form

$$N = n + s'C, \tag{2}$$

where N is intensity in counts per second, n is the counts per second for the original sample, s′ is slope of the graph, and C is the added concentration.

From figure 2 and equation (2) when $N = O$, $C = b$, and

$$b = -\frac{n}{s'} \tag{3}$$

correction of b to the X scale to give a value for X_0 may be done by substituting in equation (1) and substituting $X = O$, $C = b$.

$$O = b + X_0 (1 - b)$$

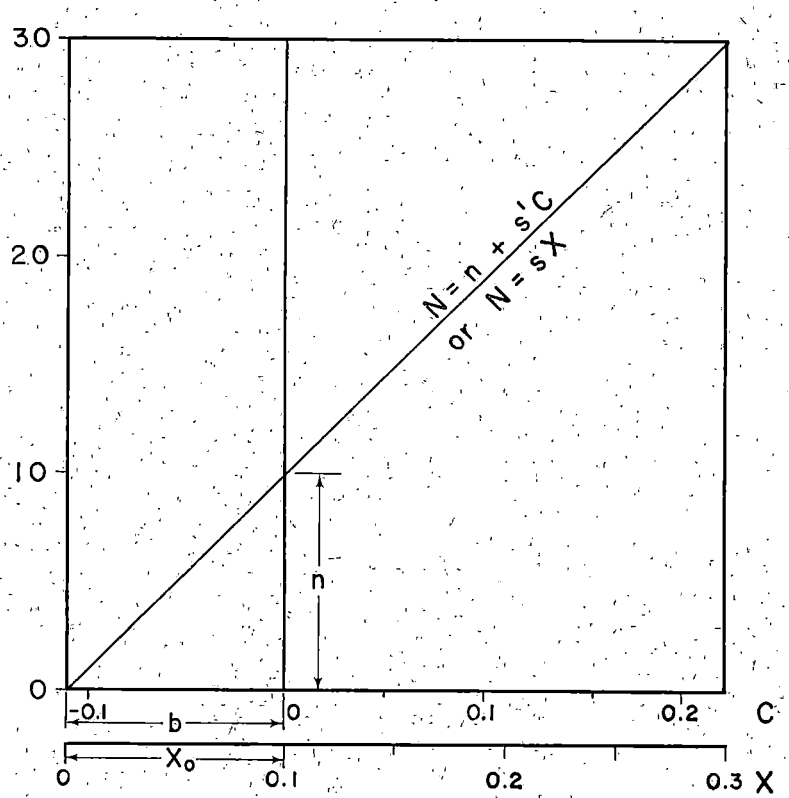

Fig. 2. Change of scale of abscissa from C, concentration added, to X, total concentration.

$$X_0 = \frac{-b}{1-b} = \frac{n}{n+s'} \tag{4}$$

Either b or s′ and n may be evaluated from a least squares fit of the data. Similarly the slope, s′, may be corrected to the X scale by equating the expressions for N and substituting for n from equation (3):

$$N = s'C + n = sX$$
$$s'C - bs' = sX$$
$$s = s' - \frac{C-b}{X} \tag{5}$$

Combining equations (1) and (4),

$$X = C - \frac{b}{1-b}(1-C)$$
$$= \frac{C(1-b) - b(1-C)}{1-b}$$
$$= \frac{C-b}{1-b} \tag{6}$$

Substituting this value in equation (5),

$$s = s'(1-b), \text{ or}$$
$$s = s' + n \tag{7}$$

In practice, s′ and n may be obtained by the least squares method to allow calculation of s from equation (7). The concentration of MnO_2 in any sample is then

$$X = \frac{N}{s},$$

or expressed as a percent,

$$P = 100\frac{N}{s}. \tag{8}$$

The resulting curve and equation for manganese are in figure 3. Because of similarity of absorption factors the equation is expected to hold reasonably well for most calcareous loess soils. Intensities from leached samples should be somewhat higher due to the removal of calcium, which is a rather effective absorber for manganese Kα (table I).

Iron

Calibration tests similar to those performed for manganese were run for iron with both Kα and Kβ radiations (table III and figure 4). Ordinarily determinations on unknowns would be by measurement of Kα, but Kβ may be used as a further check. Comparative chemical analysis and X-ray fluorescent data for two quite different loess samples are given in table 4.

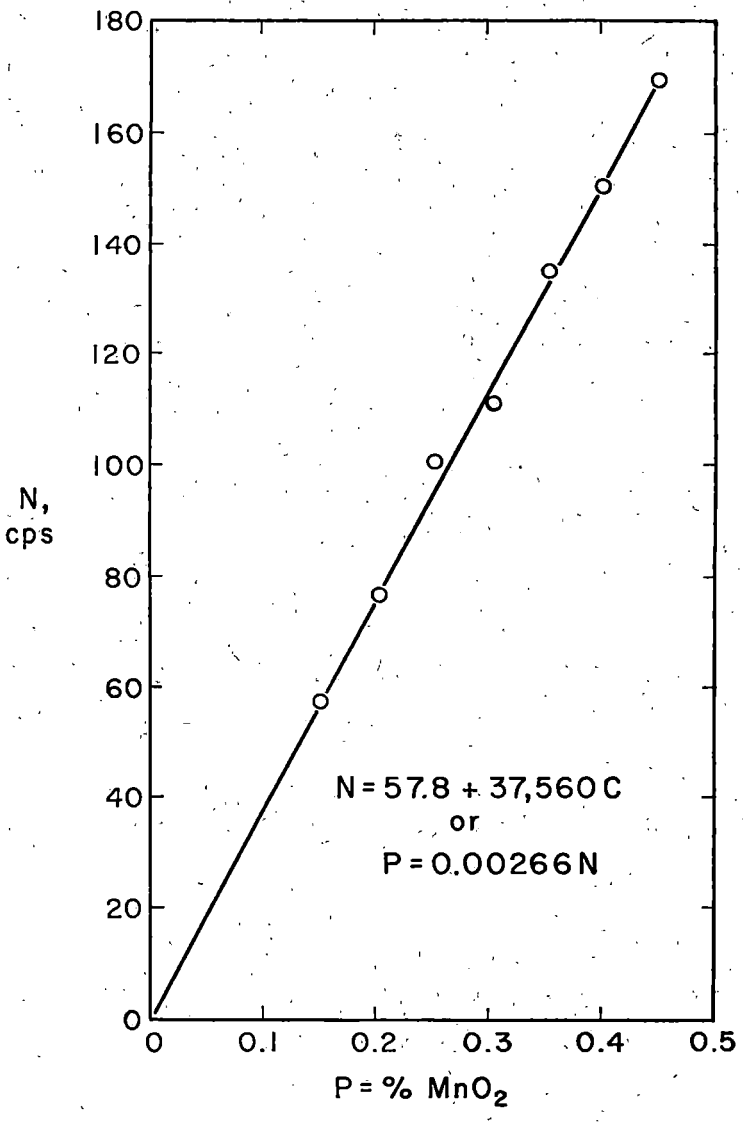

Fig. 3. Calibration curve for manganese Kα radiation (table II).

The equations of figures 3 or 4 are expected to hold true for most loess samples, but it would be desirable to apply the fluorescent method to a wider range of samples without repeating the calibration curves. For this purpose a two-point method was devised. A sample is tested for an element, then a known amount of the element is added to the sample, and the sample is retested. If a linear response is assumed, the two counting rates should be

TABLE III. IRON CALIBRATION DATA FOR A FRIABLE LOESS SOIL

% added Fe_2O_3	Total % Fe_2O_3*	FeK_α, cps†,‡ ($2\theta = 40.18°$)	FeK_β, cps†,§ ($2\theta = 36.30°$)
0	3.84	3,081	693
1.961	5.72	4,505	1,006
3.846	7.54	5,965	1,380
5.660	9.28	7,362	1,650
9.091	12.58	10,035	2,343
13.043	16.38	12,791	3,029

*0% value from least squares fit of first four points and equation 4; all others from column 1 and equation 1.

†Corrected to FeK_α count of 43,419 cps on pure powdered Fe_2O_3.

‡Background counted at $2\theta = 43.0°$, varies from 19 to 27 cps.

§Background counted at $2\theta = 35.0°$, varies from 46 to 55 cps.

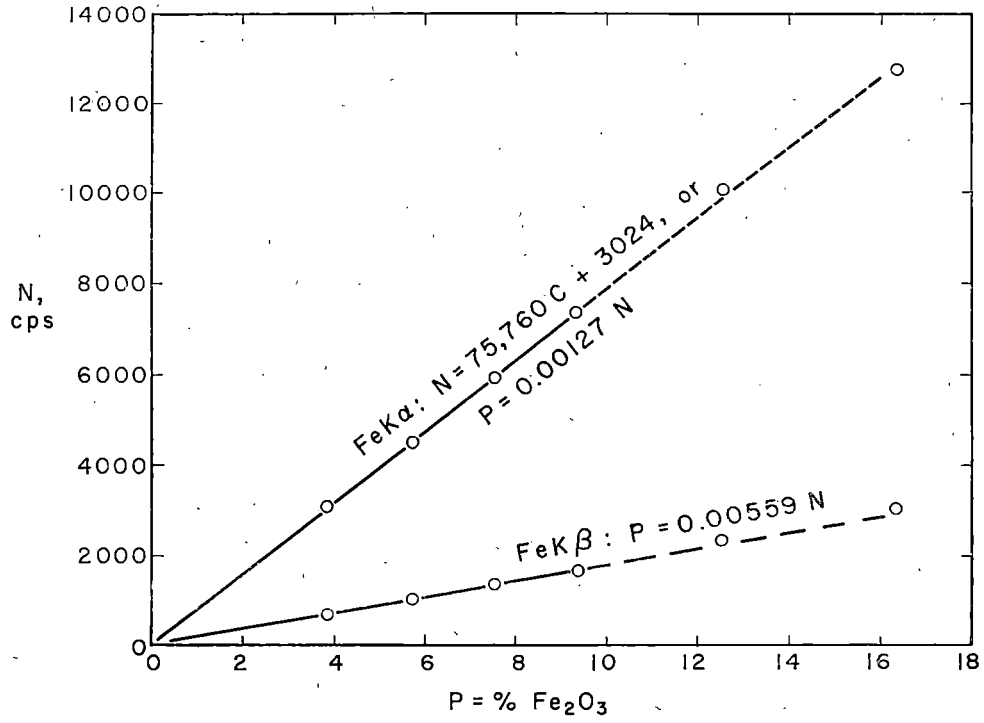

Fig. 4. Calibration curve for iron $K\alpha$ and $K\beta$ radiation (table III). Curves are fit by least squares.

sufficient to establish the slope of the response curve and allow evaluation of the content of the element in the original sample. The upper limit of the linear response was not established for manganese, but for iron it is at about 10% to 14% Fe_2O_3 (figure 4).

TABLE IV. COMPARISON OF IRON DETERMINATIONS ON TWO LOESS SOILS

Sample	FeK_α cps*	% Fe_2O_3 (Fig. 3)	% Fe_2O_3 from chemical analysis
55-1 friable, calcareous loess	1924	2.44	2.70
43½-1; plastic, leached loess	3366	4.28	4.82

*Corrected for drift and background.

If X_0 is the concentration of the element in the original sample, C is the concentration added expressed on the basis of the whole sample, N_1 and N_2 are the corresponding count rates, and a linear response is assumed,

$$\frac{N_1}{N_2} = \frac{X_0}{X} \tag{9}$$

Substituting for X from equation (1) and solving for X_0,

$$\frac{N_1}{N_2} = \frac{X_0}{C + X_0 (1 - C)}$$

$$X_0 = \frac{N_1 C}{N_2 - N_1 + N_1 C} \tag{10}$$

Use of the two-point method

Several standard samples were selected to check the validity of the two point method for determination of iron and manganese. Data are presented in table V.

TABLE V. IRON AND MANGANESE CONTENTS BY X-RAY FLUORESCENT TWO-POINT METHOD

Sample	Added % Fe_2O_3	X_0 = % Fe_2O_3	Reported % Fe_2O_3	Added % MnO_2	X_0 = % MnO_2	Reported % MnO
Nat. Bur. Std. No. 1a,* argillaceous limestone	1.511	1.659	1.63	0.385	0.071	0.038
Nat. Bur. Std. No. 56a,* phosphate rock				0.319	0.160	0.180
A.A.P.G. No. H-5,† kaolinite	0.62	0.327	1.00			
	3.95	0.320	1.00			
A.A.P.G. No. H-28,† montmorillonite	0.887	3.81	4.00			
	3.510	4.78	4.00			

*Published chemical analysis in National Bureau of Standards Circular C 398, Washington, 1946, pp. 18-19.

†Published chemical analysis in Analytical Data on Reference Clay Materials, American Petroleum Institute Project 49, New York, 1950, pp. 44-45.

The tests on an argilloceous limestone show close agreement between X-ray and chemical data for total iron, which is significant because absorption of iron $K\alpha$ by this sample is high. Manganese contents are not in close agreement, possibly due to the low content of manganese. At very low contents counting errors are magnified because of the low peak-to-peak ground ratio, and closer results might be obtained by counting for longer times. The manganese data for the sample of phosphate rock shows much closer agreement.

Two clays were analyzed for iron. One, a kaolinite, gave rather poor agreement between chemical and X-ray data, so the sample was re-run by the two-point method with a different content of added iron. The X-ray data from the two runs agree extremely well. The error may therefore be in sample differences or in chemical analysis, which may have been rounded off to one percent. Similarly, two runs were performed on a montmorillonite. They give fairly good agreement, but difficulty was experienced in mixing, which undoubtedly influenced X-ray results.

REFERENCES

Because of the newness of application, the X-ray fluorescent method has been used little for soils. Reading of a general nature plus a bibliography are in "Norelco Reporter," Vol. III, No. 2, 3, 4, 5, March-September, 1956.

EXCHANGEABLE POTASSIUM AND CLAY MINERALS IN SELECTED IOWA SOIL PROFILES

by

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ABSTRACT

Exchangeable K values and X-ray diffraction patterns were obtained for soil samples from several modal profiles of nine different soil series in Iowa. Exchangeable K in "moist" subsoils was consistently low and almost always lower than in the "moist" surface soil. Drying increased exchangeable K in all the soils but the increase due to drying varied between soil series and with depth in the profile of each series. Reversion of exchangeable K to a non-exchangeable form on rewetting the oven dry samples generally increased with depth in the profile but the exchangeable K never decreased to the original "moist" level. X-ray diffraction analyses show the presence of montmorillonite, chlorite, illite and/or mica, and kaolinite in all the profiles, with montmorillonite predominating. Vermiculite was present in some of the soil series. No relationship between the clay mineral content and the amount of exchangeable K could be established.

INTRODUCTION

Potassium (K) is held on the clay minerals in soils in exchangeable and non-exchangeable forms. The amount of exchangeable K in Iowa soils, however, depends on the moisture status of the soil[2,6]. Drying generally increases the amount of exchangeable K, especially in subsoil samples. The K released on drying may revert to a non-exchangeable form on rewetting, but the amount of reversion varies greatly between different soil samples. The amount of exchangeable K also depends on the kind and amount of clay minerals in the soil. Most of the K on montmorillonite and kaolinite is readily exchangeable, but much of the K in illite and vermiculite is not readily exchangeable because it is located between the layers of a contracted lattice. A large amount of the K in Iowa soils is presumed to be in this non-exchangeable form.

The amount of clay varies markedly between different soils and between different horizons of any one soil type found in Iowa, but little is known

about the kinds of clay minerals. On the basis of differential thermal analysis, chemical composition, and base exchange capacity in five Iowa soils (Clarion, Grundy, Tama, Webster and Weller) the average percentages of montmorillonite, illite and kaolinite were calculated to be 60, 30, and 10 respectively[5]. X-ray diffraction of the less than one micron clay, differential thermal analysis, and base exchange capacity were all used in studying different Iowa soils and the predominant mineral in Clinton, Tama, Clarion, Webster and Marshall soils was found to be montmorillonite with possibly some kaolinite and in the Marshall soil possible some illlite[4]. Only in a Gosport soil was the clay predominantly kaolinite. In an earlier study, differential thermal analysis was used to study the clay minerals in different soils[3] and it was concluded:

(1) the platy structure in certain horizons of the Clinton, Fayette, Marion and Edina soils was associated with a higher content of kaolinitic clays in these soils as compared to Clarion, Webster, Tama, and Marshall soils, which have no horizons with a platy structure;

(2) in general montmorillonite gradually increased with depth in gray brown podsols and planosols but not in prairie soils; and

(3) Lindley and Shelby soils developed on Kansan and/or Nebraskan till had a higher proportion of kaolinite than did other Iowa soils. No studies relating the amount of exchangeable K to the kinds and amounts of different clay minerals in Iowa soils have been reported.

The purpose of this study was to obtain more information concerning the kinds of clay minerals in the profiles of different Iowa soils, to determine the amount of exchangeable K in Iowa soils, and to determine if any relationship could be found between the kinds of clay minerals and the amount of the exchangeable K in these soils under different moisture conditions.

METHODS AND MATERIALS

Soil samples were collected from different horizons of modal profiles of nine different soil series. The number of profiles of each series sampled and the counties in which profiles were located are shown in table I. The soil

TABLE I. NUMBER AND LOCATIONS OF MODAL PROFILES OF DIFFERENT SOIL SERIES INCLUDED IN THIS STUDY

Soil series	Number of profiles sampled	Counties in which profiles were
Carrington	6	Mitchell, Linn
Tama	5	Marshall, Scott
Clinton	5	Jefferson
Fayette	6	Clayton, Winneshiek
Mahaska	2	Jefferson
Edina	4	Wayne, Davis, Van Buren
Shelby	3	Shelby
Marshall	3	Shelby
Weller	4	Decatur, Jefferson

samples were stored in paper cartons in a cool (40°F), constant temperature room for several months prior to analyses. During this storage the moisture content of the soil samples decreased to between 5 and 10 percent. Therefore, the soil samples were not truly field moist when analyzed, but are referred to as "moist" to distinguish them from oven dried samples.

Exchangeable K was determined on the "moist" and oven dry (110°C for 24 hours) soil samples by extracting 2 g. soil with 10 ml. neutral, 1 *N* NH_4OA_0 and determining K in the extract with a flame photometer. To determine if the K released to an exchangeable form on drying reverted to a fixed form during moist storage, 2 ml. of water was added to 1.5 g. samples of the "moist" and oven dry soils, and after 90 days storage, exchangeable K was extracted as described above.

Samples from each horizon of two of the modal profiles of each soil series were selected for X-ray diffraction analysis. Each sample was air dried and pulverized to pass a 200 mesh sieve. A representative portion of the material passing the sieve was treated with ethylene glycol to expand the montmorillonite, and packed wet into a sample holder. The surface was smoothed with a glass slide to increase preferred orientation and increase intensities of basal reflections. All analyses were made with a Geiger counter diffractometer utilizing a 1° beam slit, 0.2° detector slit, 3 second time constant, 2° per minute scan rate, 1000 cps full scale, and CuKα radiation. Selected samples were given various other treatments prior to or without glycolation, to confirm the identification of the several clay minerals.

Quantitative analysis by X-ray diffraction without the use of internal standards is at best an estimate, since diffraction intensities depend not only on the amount, composition, and crystallinity of a mineral, but also on sample packing, amount of excess glycol, degree of preferred orientation, and absorption of X-rays by the samples. With CuKα radiation absorption is particularly sensitive to the presence of iron; therefore, clay diffraction intensities may be lower in an iron-rich B horizon than in an underlying C, even though the B contains more clay mineral. Nevertheless, to summarize the X-ray diffraction data, quantitative estimates were attempted by a peak area method in which peaks were approximated by triangles. Overlapping peaks were separated and carefully evaluated with regard to symmetry and total area. Peak areas were calculated and referred to a geometric scale to give more emphasis to minor minerals. The scale is as follows:

AREA, IN.^{2}	SCALE	RATING
0	0	undetectable
<0.05	t	trace
0.05 - 0.1	1	scarce
0.11 - 0.2	2	minor
0.21 - 0.4	3	moderate

0.41 - 0.8	4	abundant
0.81 - 1.6	5	very abundant
1.61 - 3.2	6	predominant

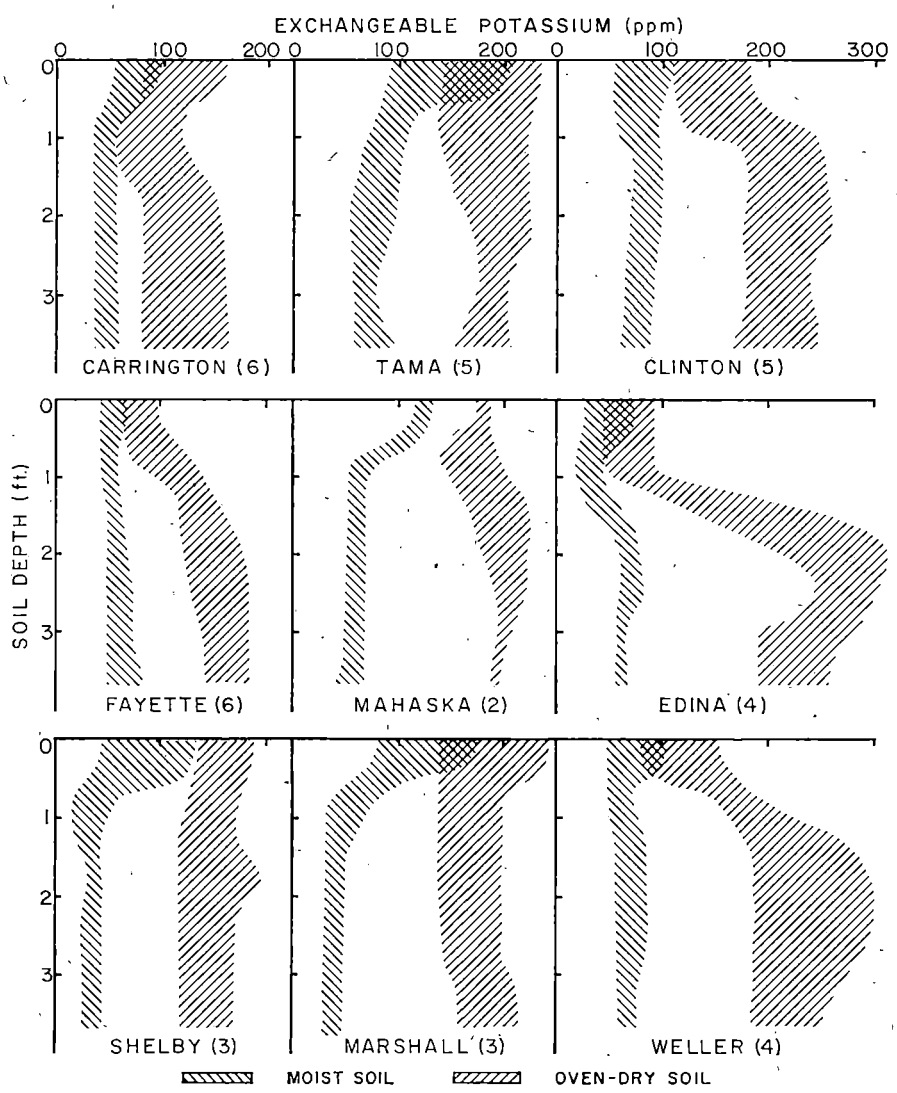

Fig. 1. Profile distribution of exchangeable K as determined on "moist" and oven-dry (110° C., 24 hours) samples of nine soil types. (Numbers in parentheses indicate the number of profiles included to establish each range in exchangeable K shown for the "moist" and oven-dry soils.)

RESULTS AND DISCUSSION

Profile Distribution of Exchangeable K

The distribution of exchangeable K in the profiles of the nine different soil series when K was extracted from the "moist" and oven dry soil samples is shown in figure 1. The ranges of exchangeable K shown in figure 1 for the "moist" and oven dry samples from the various depths include the data from all of the profiles of each soil series.

Except for the surface soil samples, the variability in exchangeable K in the "moist" samples within a soil series was relatively small. It can also be seen that the profiles of different soil series differed more than the profiles within a soil series. The "moist" K values for the surface soils did vary widely in some cases, notably in the Tama, Shelby and Marshall soils.

Exchangeable K in the "moist" samples was relatively constant with depth throughout the profiles of the Clinton, Fayette and Weller soils but decreased with depth in the Carrington, Tama, Mahaska, Shelby and Marshall soils and increased in the B horizon of the Edina profile. These data also illustrate the low level exchangeable K found in subsoils under moist conditions. Other studies have shown that these "moist" values are slightly higher than would have been obtained with truly field-moist samples. The Marshall and Shelby profile samples did not get as dry as the others, and they show more correctly that the exchangeable K in subsoils in the field is often very low, and in Iowa almost always lower than in the corresponding surface soils.

Drying increased exchangeable K in all samples, but the amount of K released on drying varied with soil type and with depth in each profile. Thus, the profile distribution of exchangeable K in each soil depended on the moisture content of the soil samples at the time of extraction of exchangeable K. Subsoil samples generally released more K on drying than did the surface soils.

Exchangeable K in the oven dry samples was relatively constant throughout the profiles of the Carrington, Tama, Mahaska, Shelby and Marshall soils (although it decreased somewhat in the A_3-B_1 horizon of the Carrington profile), but was higher in the subsoil than in the surface soil in the Clinton, Fayette, Edina and Weller profiles. All the soils in the first group have developed under grass vegetation. In the latter group, except for the Edina, the soils were developed under forest vegetation. The Edina is a highly weathered planosol developed under grass. Further studies on this relationship of exchangeable K to clay distribution in the profile and to native vegetation would, therefore, appear warranted.

Reversion of K from Exchangeable to Non-exchangeable Forms During Moist Storage.

Some of the K that has been released by drying will revert to a non-exchangeable form on rewetting[2]. It was of interest, therefore, to determine

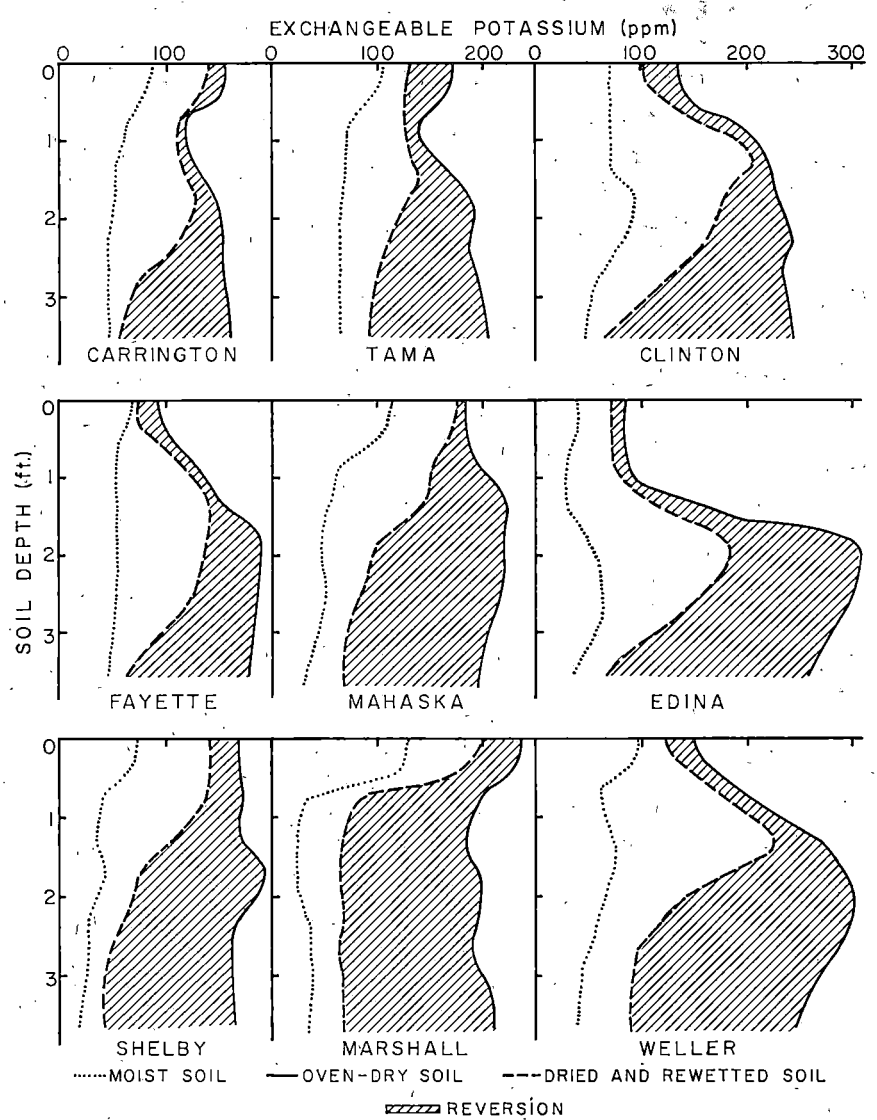

Fig. 2. Exchangeable K in oven-dry (110° C., 24 hours) and "moist" Iowa soil profile samples that have been stored in water for 90 days. The exchangeable K values obtained with the oven-dry samples are also shown for comparison.

if the amount of reversion varied with depth in the profile and from one soil series to another. The amount of reversion that occurred when oven dry samples from selected profiles of the different soil series were stored in water for 90 days was determined (figure 2). The exchangeable K values obtained after rewetting the "moist" samples are shown by dotted lines for comparison.

It can be observed, by a comparison of the "moist" values in figures 1 and 2, that there was little change in exchangeable K in these "moist" samples due to storage in water for 90 days. Although some release of K had occurred in these "moist" soil samples that were partially dried, rewetting appeared to result in very little reversion of this K to a non-exchangeable form.

In most of the soil profiles there was only a small amount of reversion in oven dry samples from the upper horizons. The amount of reversion increased with depth in the profile, but reversion did not decrease the exchangeable K level to that found in the "moist" soil. The profile pattern of reversion varied markedly from one soil series to another. These results indicate that the process of K release on drying is only partially reversible and that rewetting of dried soil samples, especially from the surface horizon, will not result in exchangeable K values similar to those obtained for undried soils.

X-Ray Diffraction Patterns for Profile Samples

X-ray diffraction patterns were obtained for the various profile samples to provide information concerning the kinds and relative amounts of different clay minerals. The relative intensities of the X-ray diffraction peaks indicate 17 *A*, 14 *A*, 10 *A*, and 7 *A* spacings in glycolated samples (table II). X-ray diffraction patterns for the different horizons of a Marshall silt loam profile (No. 39) are shown in figure 3. The relative intensity ratings of different peaks are shown in this figure to illustrate the size of peaks represented by the data in table II.

Relative intensity ratings for samples from similar horizons of different profiles of the same soil series usually agree within one scale unit and never differ by more than two units. The relative intensities of the peaks tend to be lower and more variable for the A horizon samples than for the subsoil samples. Interferences as a result of the higher organic matter contents of the A horizons could be responsible for much of this variability and lower intensities.

The largest diffraction peak for nearly all glycolated samples was in the neighborhood of a 17.7 *A* spacing. In untreated samples heated to 700° C. for 0.5 hour this 17 *A* peak was replaced by a large 10 *A* and a smaller 9.6 *A*

TABLE II. RELATIVE INTENSITIES OF X-RAY DIFFRACTION PEAKS FOR SAMPLES FROM PROFILES OF DIFFERENT SOIL SERIES IN IOWA

Soil series Profile		Sampling depth (inches)		Genetic horizon		Relative intensities*								Mixed interlayer spacing (A)	
						17 A		14 A		10 A		7 A			
I	II	I	II	I	II	I	II	I	II	I	II	I	II	I	II
		0	0												
Carrington				A_1	A_p	i	4	2	1	1	2	t	2	16,13	....
13,	15	6	6												
				A_3-B_1	A_3-B_1	1	3	2	1	1	2	1	t	20,12	12
		14	12												
				B_2	A_3-B_1	4	3	3	2	1	1	2	1	....	....
		26	18												
				B_2	B_2	3	3	3	2	2	t	1	t	....	12
		27	24												
					B_3		4		2		t		1	....	....
			30	C		3		1		1		1			
					C		4		1		1		1		....
		36	36												
				C	C	4	5	2	2	1	t	3	2	....	....
		48	48												
		0	0												
Tama				A_p	A_p	1	1	t	1	1	1	t	t	16,13,11	12
1,	6	6	6												
				A_3	A_3	2	2	1	1	1	1	t	t	20,16,12	13
		13	15												
				B_2	B_2	3	3	3	1	2	1	1	t	....	....
		19	20												
				B_2	B_2	4	4	2	3	2	1	1	t	20	....
		25	26												
				B_3	B_3	4	4	3	3	1	t	t	1	24	....
		31	31												
				C	C	4	4	3	2	1	1	1	1	11	11
		37	42												
				C		4		3		2		1		24	
		49													
		0	0												
Clinton				A_p	A_p-A_2	2	3	1	2	0	2	t	t	....	12
7,	9	5	6												
				A_2	B_1	4	4	3	3	1	1	1	2	....	24
		12	12												
				B_1	B_1	4	4	2	3	1	1	0	t	....	....
		18	18												
				B_2	B_2	4	5	2	2	t	1	t	1	....	....
		26	24												
					B_2		4		2		t		1		....
			30	B_3		4		2		1		t		....	
					B_3		4		3		1		t		....
		36	36												
		36	36												

TABLE II. (CONTINUED)

Soil series Profile I	Soil series Profile II	Sampling depth (inches) I	Sampling depth (inches) II	Genetic horizon I	Genetic horizon II	Relative intensities* 17 A I	17 A II	14 A I	14 A II	10 A I	10 A II	7 A I	7 A II	Mixed interlayer spacing (A) I	Mixed interlayer spacing (A) II
Clinton				B_3		4		2		1		1		....	
7,	9	42			B_3-C		5		3		2		t		....
				C		4		2		1		t		....	
		48	48												
		0	0												
Fayette				A_p-A_2	A_p-A_2	t	1	1	t	1	1	t	t	12	16,13,11
20,	22	6	6												
				B_1	B_1	2	3	1	2	1	1	t	t	16,12.5	....
		12	12												
				B_1	B_1	3	3	2	2	1	t	t	t	24	....
		18	18												
				B_2	B_2	5	4	3	3	1	t	1	t	....	....
		24	24												
				B_2	B_2	5	4	4	3	2	1	1	t	....	....
		36	36												
		48	48	B_3	B_3	4	4	3	3	1	1	t	1	12	....
		0	0												
Mahaska				A_p	A_p	3	1	t	t	1	t	0	t	....	....
28,	29	8	6												
				A_3-B_1	A_3-B_1	4	3	t	1	t	2	t	t	12	....
		13	13												
				B_2	B_2	4	5	1	t	t	2	0	1	....	24
		19	19												
				B_2	B_3	4	5	1	1	t	2	t	1	....	24
		26	26												
				B_3	C	4	5	1	2	t	2	t	t	....	....
		33	36												
				C		4		2		t		1		24	
		40	48		C		5		2		2		1		....
				C		4		t		1		t		....	
		0	0												
Edina				A_p	A_p	2	2	1	t	1	1	t	t	....	24,12
27,	32	8	8												
				A_2	A_2	2	2	t	t	1	2	t	t	....	....
		15	14												
				B_2	A_2	5	3	1	t	t	t	t	0	....	....
		22	19												
				B_3	B_2	5	5	2	t	1	1	t	t	12	....
		29	28												
				C	B_3	5	5	1	t	t	t	t	t	....	....
		36	37												
				C	C	5	5	3	t	t	t	t	t	....	....
		48	48												
		0	0												

TABLE II. (CONTINUED)

Soil series Profile I	Soil series Profile II	Sampling depth (inches) I	Sampling depth (inches) II	Genetic horizon I	Genetic horizon II	Relative intensities* 17 A I	17 A II	14 A I	14 A II	10 A I	10 A II	7 A I	7 A II	Mixed interlayer spacing (A) I	Mixed interlayer spacing (A) II
Shelby				A_p	A_p	4	3	1	t	2	1	t	t	....	12
37,	38	6	6												
				B_2	B_1	5	3	t	t	1	1	1	1	....	....
		12	12												
				B_2	B_2	5	4	3	1	t	1	1	2	....	12
		18	18												
				B_2	B_2	4	5	2	1	1	2	t	2	12	24
		24	24												
				B_2		4		3		1		1		....	
		30			BC		3		t		2		1		12
				B_3		4		2		2		1		12	
		36	33												
				C	C	4	2	3	t	2	2	1	1	....	....
		42	39												
				C	C	4	4	0	1	1	1	2	1	....	11
		48	48												
		0	0												
Marshall				A_p	A_p	3	3	1	1	2	2	t	1	....	12
34,	39	6	6												
				B_1	A_3	4	4	3	2	1	1	1	t	....	13
		12	12												
				B_2	B_2	5	4	3	2	2	2	1	1	....	12
		18	18												
				B_2	B_2	5	4	2	3	2	2	1	t	....	12
		24	24												
				B_2	B_2	5	4	3	3	1	1	1	t	12	....
		30	30												
				B_3	B_3	5	5	3	4	2	1	1	1	....	....
		36	36												
				C	C	5	5	3	3	2	2	1	1	....	11
		42	42												
				C	C	5	5	3	3	2	3	1	1	....	....
		48	48												
		0	0												
Weller				A_p	A_p	2	1	1	1	1	1	t	t	12	....
Decatur Co.		6	5												
24,	25			A_2-B_1	A_2-B_1	4	3	1	2	2	1	t	1	....	....
		11	10												
				B_2	B_2	5	5	1	2	2	1	t	t	24,12	....
		20	20												
				B_3	B_3	5	5	1	t	1	1	1	0	12	....
		24	29												
				B_3-C	C	6	4	1	2	1	1	0	t	12	....
		36	36												
				C	C	5	5	1	1	1	1	t	1	11	20
		48	48												
		0	0												

TABLE II. (CONTINUED)

Soil series Profile I	Soil series Profile II	Sampling depth (inches) I	Sampling depth (inches) II	Genetic horizon I	Genetic horizon II	Relative intensities* 17 A I	17 A II	14 A I	14 A II	10 A I	10 A II	7 A I	7 A II	Mixed interlayer spacing (A) I	Mixed interlayer spacing (A) II
Weller				A_p	A_p	i	3	1	1	t	1	t	t	16	12
Jefferson Co.		7	6												
				A_2	A_2	4	5	2	1	2	1	t	t	20,11	....
30,	'33	15	13												
				B_2	B_2	5	5	2	2	2	1	t	t	11	24,12
		23	22												
					B_3		5		2		2		t		....
			32	B_3		5		2		2		t		....	
					C		5		2		1		t		....
		36	40												
				C	C	5	5	2	3	1	t	t	t	....	....
		48	48												

*0—No detectable peak
t—trace
1—scarce
2—minor
3—moderate
4—abundant
5—very abundant
6—predominant

i—all in interlayer

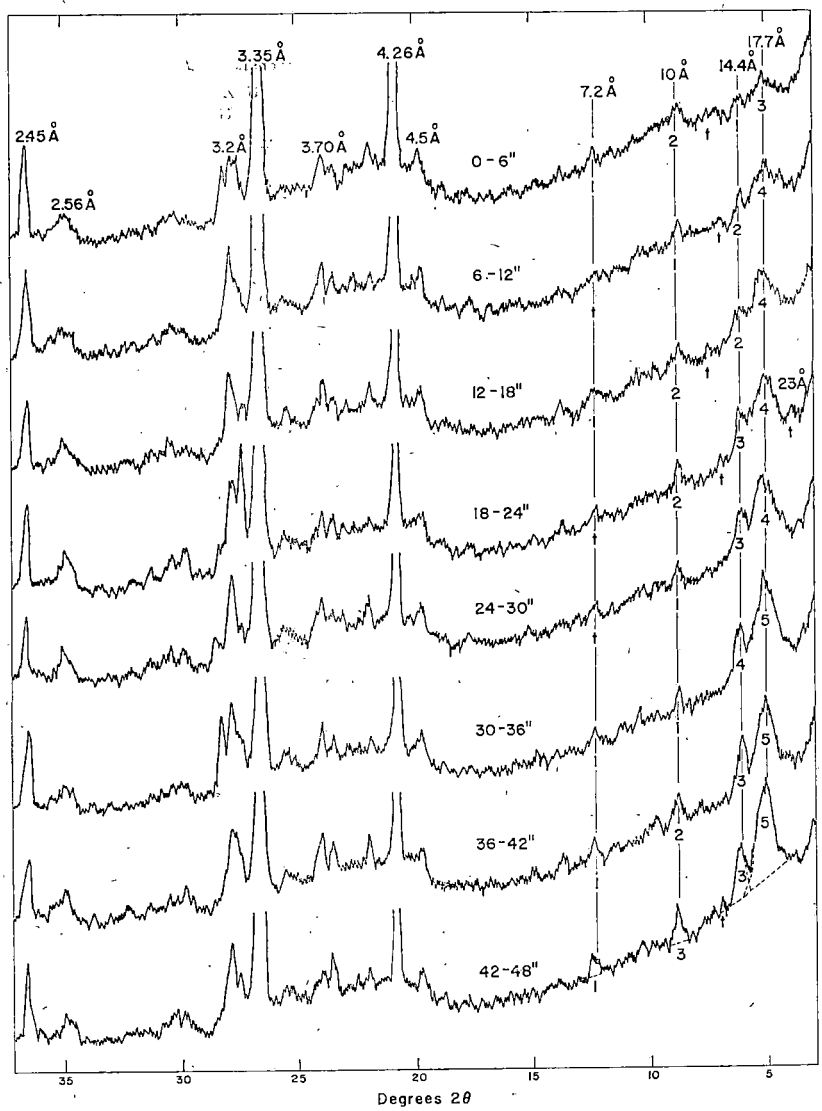

Fig. 3. X-ray diffraction patterns for glycolated samples from different horizons of a Marshall silt loam profile (profile No. 39).

peak. These results indicate that a montmorillonite-type mineral is in all these soil profiles. Broadness of this peak suggests a very fine crystallite size in the C-axis direction.

Relative intensity ratings of 2 to 6 were obtained for the 17 A peaks in the B and C horizons of all profiles, with ratings of 4 and 5 predominating.

This indicates an abundant amount of montmorillonite in the parent material and in the zone of clay accumulation. The relative intensities of 17 *A* peaks for samples from A horizons were lower and more variable, ranging from trace to 5.

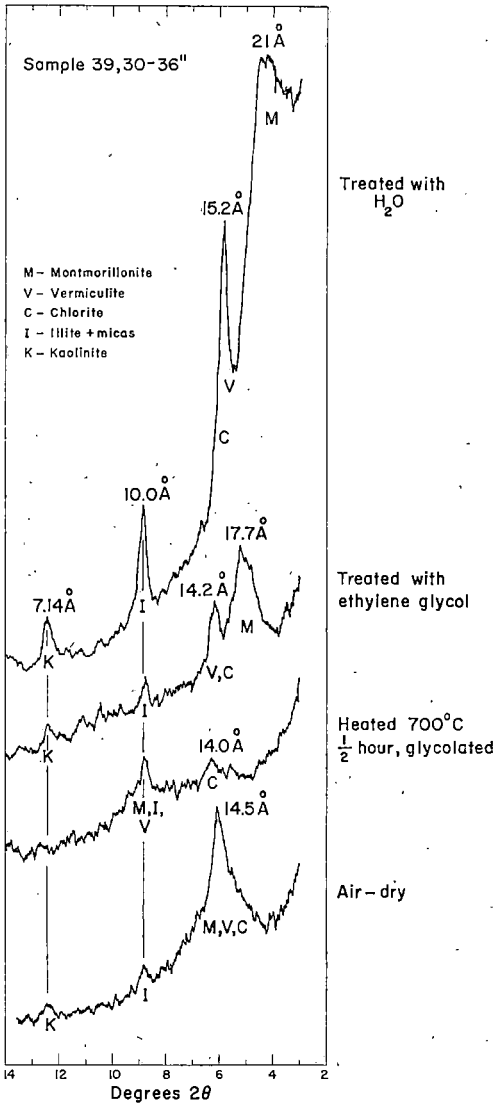

Fig. 4. X-ray diffraction patterns comparing the effects of different treatments on the 30 to 36 inch depth sample from a Marshall silt loam profile (No. 39).

Most of the profile samples gave a diffraction peak indicating a spacing of 14.4 to 14.0 A. This peak could be due to chlorite and/or vermiculite. Therefore, fresh material from about one-third of the samples was heat-treated at 700° C. for 0.5 hour and re-run. Heat treatment reduced the 14 A peak, but failed to eradicate it, indicating the presence of chlorite (figure 4). Wetting a fresh sample with water usually removed the 14 A peak or masked it with a strong, sharp 15 A peak. Calcium-saturated vermiculite immersed in water has an 001 spacing of 15 A[1]. This 15 A peak was not observed in soil samples (such as Edina 32 A) that did not exhibit a 14 A peak when treated with glycol. Thus, the 14 A peak probably indicates vermiculite plus trace amounts of chlorite. Intensities of the 15 A and other clay mineral peaks in the water-wet samples were considerably increased by preferred orientation. Sharpest peaks occurred after setting a few minutes.

The 14 A peaks were generally highest in samples from the B and C horizons. Very low intensity 14 A peaks were observed for almost all surface soil samples. The 14 A peaks were especially prominent in B and C horizon samples from the Fayette and Marshall profiles, with intensity ratings of 3 and 4. The Mahaska, Edina and Weller profiles had only minor 14 A peaks (except for a single C horizon samples from one Edina and one Weller profile), and B and C horizon samples from the other profiles generally gave intensity ratings of 2 or 3. One Shelby profile had consistently larger peaks than the other.

Small peaks indicating a 10 A spacing characteristic of micas and illite were common. No attempt was made to distinguish these minerals. Probably both classes of minerals are present, but apparently in small amounts, since low relative intensity ratings of 1 and 2 predominate.

There was a small diffraction peak at a spacing of 7 A in most of the samples. This spacing may be due to kaolinite or a second order reflection from chlorite. The 7 A peak disappeared when the samples were heated at 700° C. This means that kaolinite was present, and the chlorite (indicated by 14 A spacing, above) is probably a low iron variety. The relative intensities of the 7 A peaks for samples from these profiles was consistently low—generally only a trace. Only one C horizon sample, from a Carrington profile, had a rating as high as 3.

In some samples a small degree of random interlayering was indicated by small, broad X-ray diffraction peaks at several spacings. Most commonly these spacings were in the neighborhood of 11 to 12 A; however, others occurred at 13, 16, 20 and 22 - 24 A. Indications of interlayering were present for fewer than half of the samples, and, where present, the relative intensity rating usually showed only a trace. Interlayering or incomplete expansion of the montmorillonite was most pronounced in several A horizon samples.

Other minerals indicated by X-ray traces in figure 3 include quartz, which gave large, off-scale peaks at 4.21 *A* and 3.35 *A* and a sharp peak at 2.45 *A*. A smaller, apparent 3.70 *A* peak was the Kβ reflection from the 3.35 *A* quartz spacing (the filter thickness selected eliminated 95 percent of the Kβ radiation). Sharp single, double or triple peaks in the neighborhood of 3.2 - 3.4 *A* are attributed to feldspars. Other broader peaks at about 4.5 *A*, 3.5 *A* and 2.5 *A* are non-diagnostic diffractions from the clay minerals.

GENERAL DISCUSSION AND CONCLUSIONS

The data from several modal profiles of each soil series indicate that the exchangeable K content and the effect of drying on the exchangeable K content are characteristic for each soil series. In undried samples the exchangeable K contents are higher and more variable in the surface soil than in the subsoil, and are consistently very low in the subsoil horizon. Drying increased exchangeable K in all of the soil samples, but the increase was generally much greater in subsoil than in surface soil samples. Reversion of K from exchangeable to non-exchangeable forms on rewetting occurred in all samples, but the amount of reversion generally increased with depth in the profile.

The X-ray diffraction analyses indicate that montmorillonite, chlorite, illite or mica, and kaolinite were in all of the profiles. The predominating micaceous mineral in all samples, however, was montmorillonite. Vermiculite was in some of the soil series. This agrees with the results of earlier investigators, but they did not consider vermiculite in their studies. The X-ray diffraction patterns for different profiles of any one soil series were very similar. The greatest difference between soil series was in the relative intensities of the 14 *A* peaks obtained for samples from the B and C horizons. Soils of the Marshall and Fayette series had 14 *A* peaks of high intensity, whereas soils of the Mahaska, Edina and Weller series had 14 *A* peaks of low intensity.

The X-ray data indicate differences in the amounts of montmorillonite, vermiculite, and mica in the various profile samples; but it was not possible to develop a relationship between the clay mineral composition and the exchangeable K in the profiles. The differences in relative intensities of the 14 *A* (vermiculite) peak between different soil series does not appear to be related to any of the differences in amounts of exchangeable K in these profiles. Differences between surface and subsoil samples were observed in both the X-ray diffraction patterns and exchangeable K, but no consistent relationship was evident.

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NOTES ON DETERMINATION OF CARBONATES IN SOILS BY CHEMICAL MEANS, D.T.A., AND X-RAY

by

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(Iowa Academy of Science Proceedings, 66:283-301. 1959.)

ABSTRACT

Of the various methods studied for measuring carbonates in soils gas evolution methods are accurate but slow and susceptible to experimental error. Acid neutralization is rapid and offers a fair approximation, but it is affected by many non-carbonate minerals. Versenate methods are rapid and accurate, but they are affected by exchangeable calcium on the clay. None of these methods is ordinarily specific for calcite or dolomite.

Differential thermal analysis is accurate and specific for calcite or dolomite. Best results were obtained by measuring peak height after runs in an air atmosphere. However, analyses are slow and are influenced by changes in the apparatus.

X-ray diffraction from a copper tube is accurate if peak area rather than peak height is measured, but the intensities are sensitive to the presence of iron, which absorbs most of the X-rays. X-ray fluorescence measurement of calcium $K\alpha$ radiation is accurate and less susceptible to variable absorption. But since this method is affected by non-carbonate calcium, correction by leaching and re-testing is suggested. This appears to be the method most worthy of further examination.

The content of carbonate minerals in soils and unconsolidated sediments is of interest not only because carbonates indicate ancient and modern weathering zones and regional variations in unweathered material[8], but also because of the effects on soil properties. Only a trace of carbonates influence pH, identity of exchangeable cations, plasticity, and potential use of a soil. Also carbonates are important in acid methods of engineering soil stabilization, for example with phosphoric acid[2] or lignins[4], since carbonate minerals react with acidic stabilizers and either increase or ruin their effectiveness. A method for rapid, precise measurement of carbonates, especially in the low content range, is therefore of prime importance.

METHODS

Several methods for carbonate determination are used; the plan of this paper is first to discuss these methods in general terms and point out their relative merits and disadvantages, and to present data on the search for a new method via differential thermal analysis, or X-ray fluorescent or diffraction techniques.

Chemical Methods

Several methods for chemically determining the amounts of carbonates in soils are used; unless accessory tests are used, these methods are nonspecific for calcite ($CaCO_3$), dolomite, $CaMg(CO_3)_2$, or any other soluble carbonate. Results are usually calculated and reported as percent $CaCO_3$. The methods all utilize dissociation of carbonates by acid:

$$CaCO_3 + 2H^+ \rightarrow Ca^{++} + H_2O + CO_2 \uparrow$$

Chemical methods fall into three categories, depending on whether they measure the number of hydrogen ions used up (acid neutralization), the amount of CO_2 gas evolved, or the number of calcium ions released in solution.

Gas evolution. Theoretically the most accurate methods for carbonate measurement involve treatment of a soil with excess hydrochloric acid and measurement of the CO_2 gas evolved. In fact, field identifications are usually made on this basis, the presence of 0.5% or more carbonate being indicated by the sight or sound of fizzing[15]. In the laboratory the amount of gas evolved is measured volumetrically with a gas burette or gravimetrically, for example by bubbling the CO_2 over soda-lime ($NaOH + CaO$)[6], or by weight loss in the sample[15].

For greatest accuracy these methods are slow and exacting, since they are susceptible to experimental error. Volumetric measurement of the gas requires correction for temperature and barometric pressure. In gravimetric measurement all HCl and water vapors must be trapped, and precise weighing is necessary to detect minute changes due to CO_2. The reaction and evolution of gas from soils often requires 24 hours. Finally there is the problem of air initially dissolved in the acid and adsorbed on the soil mineral surfaces and CO_2 dissolved and adsorbed after the reaction takes place. These can contribute to the error, e.g. samples 21-2 and 32-1 (table I). Air is eliminated from the measurement by methods employing selective absorption. Error from dissolved CO_2 may be reduced by heating[6].

Acid neutralization. Measurement of CO_2 gas evolved is at best slow but sure, but it is not adapted to routine analysis. A far more rapid but theoretically less exact method is by acid neutralization, which means treating soil with an excess of hydrochloric acid and back-titrating to determine how much acid is used[15]. This method is the one most widely used in soils work.

TABLE I. CARBONATE CONTENTS OF LOESS AND LOESSIAL SOILS

Sample No.	% 2μ clay	pH	% $CaCO_3$ by indicated method				
			CO_2	HCl	Versenate	DTA peak ht.	X-ray Fluor.
Calcareous samples							
16-1			11.56	12.00			
16-2			4.86	3.90			
18-1			11.39	13.62			
21-2			13.56	6.55			
33-1			13.62	11.50			
55-1	10.0	8.0	11.0		7.57		
49-2				16.2		10.4	11.4 ± 0.5
22-1				15.0		9.4	11.5 ± 0.6
23-1				12.3		7.7	9.3
24-1				11.8		7.4	9.0 ± 0.7
Leached samples							
26-1	21.5	7.0		1.37			
30-1	29.5	6.4		1.62			
32-1			9.75	1.00			
36-1	28.9	6.6		1.75	0.73		1.6 ± 0.4
46-1				1.50	0.89		
100-1					2.8		2.4 ± 0.3
100-2					2.3		1.6 ± 0.4
119-5		5.7			1.8		0.9 ± 0.5
502-2	42.0	5.2			0.44		0.9 ± 0.5

The major weakness of the acid neutralization method is the assumption that all acid is used by carbonates. Other minerals, such as gypsum, hydrous iron oxides, and clay minerals, are also soluble or partly soluble in HCl. For example, dissolution of the hydrous iron oxide minerals goethite or lepidocrocite will mean:

$$Fe_2O_3{\cdot}H_2O + 6H^+ \rightarrow 2\,Fe^{+3} + 4H_2O,$$

which uses up acid. A soil containing 2.0 percent by weight acid-soluble iron hydroxide and no carbonate will thus be erroneously reported as having 3.4 percent $CaCO_3$.

Versenate. To overcome some of the errors inherent in techniques involving measurement of acid neutralization, versenate solutions may be employed to specifically measure Ca^{++} and Mg^{++} in the acid leachate. Versenate methods are rapid, and results are reproduceable[5]. Unfortunately several sources for error remain. The presence of soluble noncarbonate Ca^{++} and Mg^{++} contributes error; in soils the most important source of this error is the exchangeable calcium held by the clay. Soils described in the field as leached (table I) and with a naturally acid pH, which automatically negates the possibility for carbonates, may show one-half to two or more percent carbonate by the versenate method. Acid neutralization similarly shows about 1.5 percent erroneous carbonate. A correction may be applied to versenate results if the cation exchange capacity and identity of the exchange-

able cations are known, but the validity remains questionable due to the different conditions in which the exchange takes place. Also these measurements require much much more testing.

Calcite vs. dolomite. As previously mentioned, the usual techniques for carbonate measurement in soils do not involve separation of calcium and calcium-magnesium carbonates, and results are reported as percent $CaCO_3$. Thus the presence of 5.0 percent dolomite, $Ca{\cdot}Mg\ (CO_3)_2$ will be reported as 5.5 percent $CaCO_3$.

DIFFERENTIAL THERMAL ANALYSIS

Apparatus and Method

DTA is used for qualitative identification of clay minerals and carbonates in soils, and is recognized as a tool for qualitative analysis. In this method a sample is heated at a constant rate and differences in temperature between the sample and a sample of inert material heated in an identical manner are measured continually. The method is applicable to carbonates because of the calcination reaction.

$$CaCO_3 \rightleftarrows CaO + CO_2 \uparrow + \Delta H$$

ΔH is negative and ordinarily contributes an endothermic reaction peak at 700 - 900° C. (figure 1). Dolomite contributes two peaks, the first signifying breakdown of $MgCO_3$. This research was limited to studies with calcite.

Vertical furnaces were hung from the ceiling and counterbalanced for ease in lifting. An automatic heating rate controller provided for a rate of 10° C. per minute. The sample block of 18-8 stainless steel lay flat and was ¾″ high by 1¾″ in diameter. Two vertical ⅜″ by ½″ deep sample holes were symmetrically located with centers one inch apart. No. 22 Pt-Pt 10% Rh thermocouples were used. The furnace temperature couple was a separate chromel-alumel junction inserted in a ⅜″ d. by ½″ deep hole drilled up into the bottom of the block. The block was supported by a hollow ceramic pedestal.

Reactions

Factors affecting the size and shape of a DTA peak include the rate of heating (12, 14). More rapid heating gives sharper peaks and may affect peak area. This factor is therefore controlled.

The DTA peak is affected by the rate of reaction, i.e., reaction kinetics[9, 10]. These are not readily mathematically predictable, but for one soil type they should be systematic so that if percent reactant is plotted against peak height or area, a smooth curve, but not necessarily a straight line, will result.

The peak will be affected by heat balance in the specimen, which depends

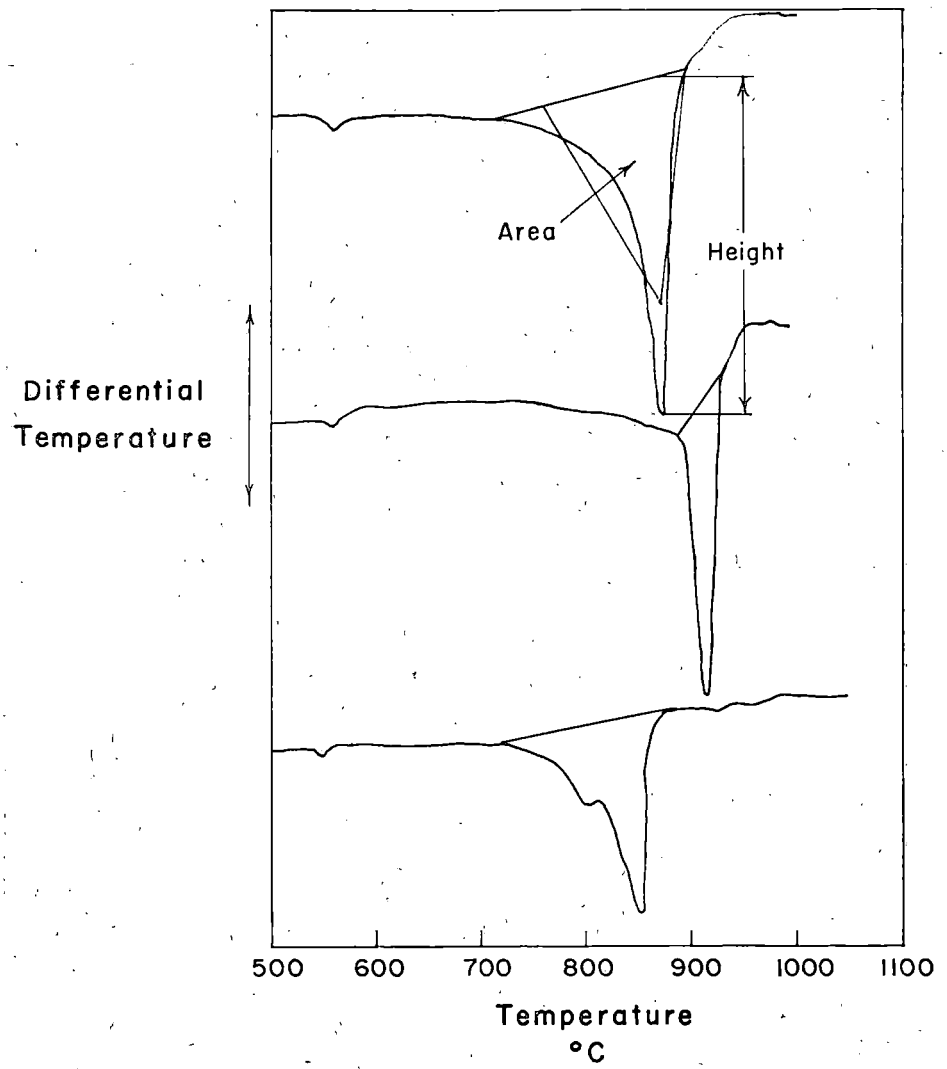

Fig. 1. Typical DTA curves for calcareous soils. Labels on the upper curve show methods for measuring peak height and area. Middle curve was run with a CO_2 atmosphere, which delays the carbonate reaction. Lower curve shows the presence of dolomite.

on thermal conductivities and specific heats within the sample[13, 14] and the composition of the atmosphere. Conductivities and specific heats are affected by simple particle size and packing, and will change during the reactions. These variables therefore defy mathematical treatment; but since they are systematic, they should give smooth empirical relationships. Composition of the atmosphere should not affect simple transition reactions, for example

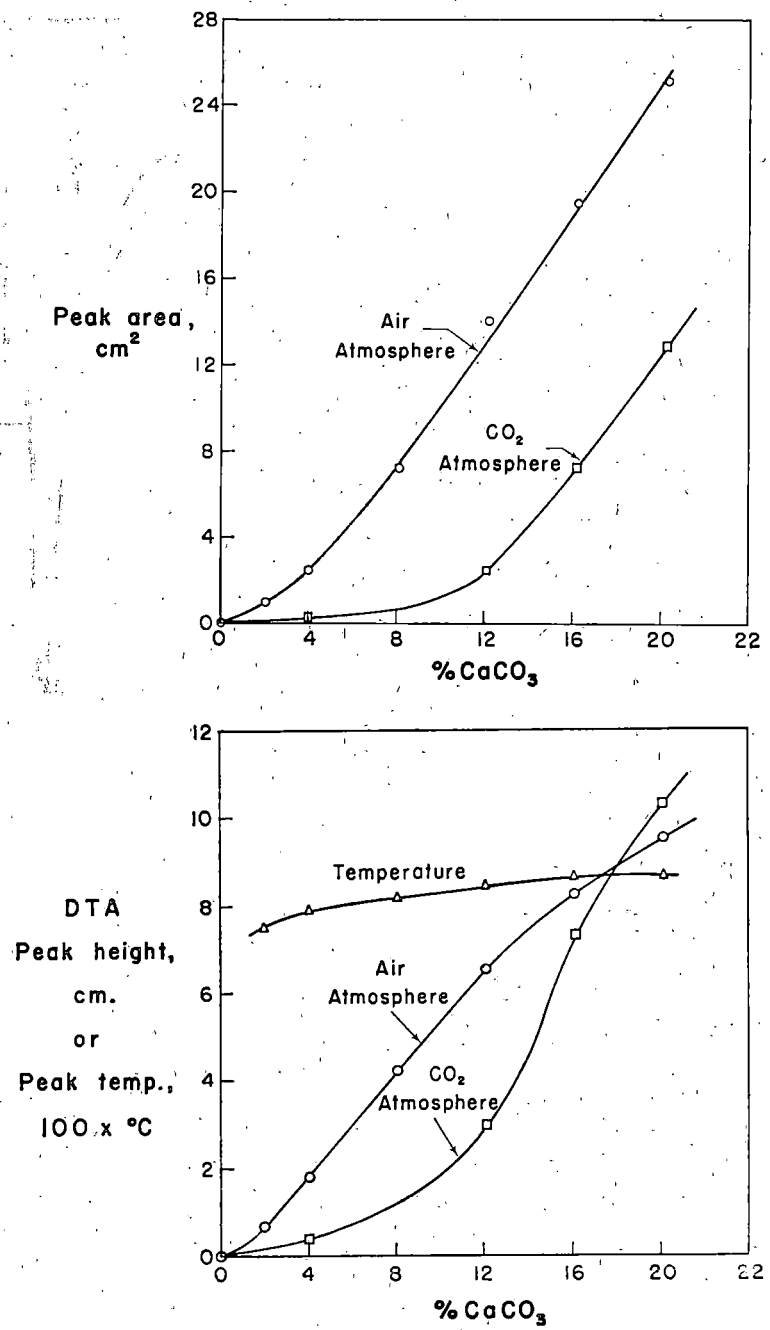

Fig. 2. Top graph shows relationship between percent calcite and DTA peak area from prepared samples. Lower graph shows percent calcite vs. peak height for an air atmosphere and for a CO_2 atmosphere, and shows the shift in peak temperature for runs in air.

the quartz inversion at 573°C, but it strongly influences the carbonate reaction. If thermal conductivity, specific heat, and influence of atmosphere and reaction rate all could be held constant, linear relationships between peak area and concentration of reactant would be expected[13].

Experimental Procedures

Artificial mixes. Since DTA peak parameters are expected to have systematic relationships with concentration of reactants, other factors being held constant, a friable calcareous loess soil from Harrison County, Iowa, (sample 20-2VII) was leached with HCl, re-enriched with different amounts of C.P. calcium carbonate and run by DTA. Thermocouple junctions had previously been accurately centered in the sample holes, and a uniform hand packing procedure was adopted. Analyses were run in air and in CO_2 at one atmosphere.

Typical DTA curves on these mixtures are shown in figure 1. Peak heights and areas were measured as shown. These are plotted against percent carbonate in Figure 2.

In air the reactions begin at about 700°C and reach a peak at a temperature related to carbonate content (figure 2a). Thermal lag within the sample caused by heat used by the reaction is insufficient to explain this gradual peak displacement, which reaches a maximum of 90°C. This suggests that CO_2 released from the reaction builds up a partial pressure and acts as a

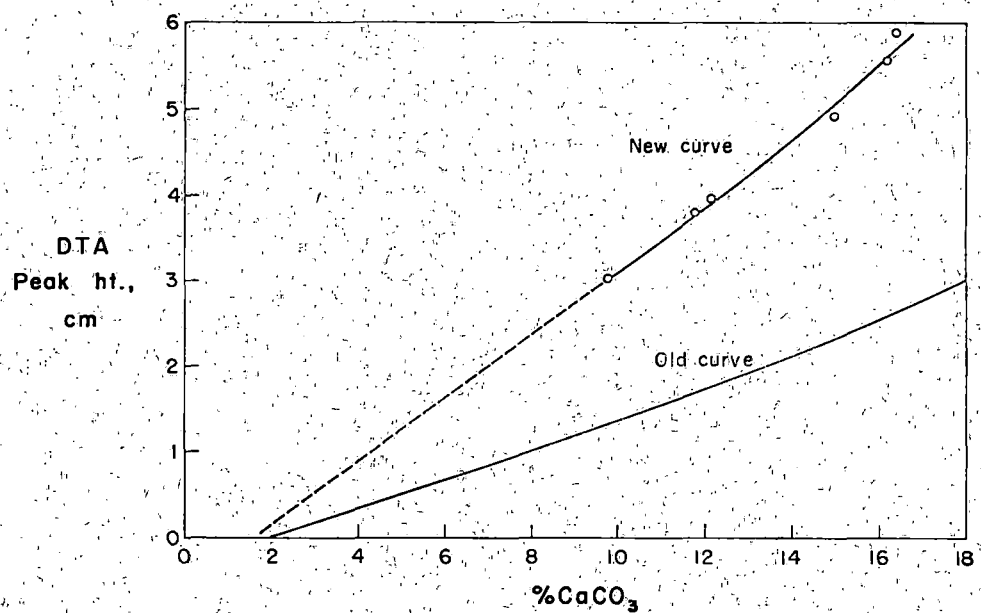

Fig. 3. DTA calcium carbonate calibrations for natural soils. New curve resulted from a thermocouple re-alignment.

retarder. Since this in turn undoubtedly affects ΔH, quantitative relationships are not expected to be linear (figure 3).

It was therefore decided to try to control the atmosphere by introducing CO_2, a decision also reached by many other researchers. In a CO_2 atmosphere the decomposition reaction does not begin until the temperature is about 830°C. Peak height and area are greatly reduced, a further indication that ΔH varies considerably depending on reaction temperature and environment (figures 1, 2). At high carbonate contents the plotted relationships for runs in air and in CO_2 more closely match, further suggesting that CO_2 pressures become significant even when the only source for CO_2 is the reaction itself.

The CO_2 atmosphere in general decreases the linearity of plotted relationships, particularly in the low carbonate content range. Several possibilities may be suggested: perhaps CO_2 did not permeat the sample adequately, or perhaps small differences in temperature are muffled by heat transfer from the flowing gas. Rather than attempt to use gas pressure techniques, use of CO_2 was discontinued.

Natural soils. Non-linearity and lack of a precise mathematical relationship for the plotted curves prevent use of equations with added amounts of carbonates as internal standards, as in X-ray fluorescence analysis. The only method remaining was preparation of empirical calibration curves (figure 2). The noncarbonate matrix was a single loess soil; it was next decided to see if the same curve might hold when the matrix varies.

A plot of peak height vs. carbonate content of different natural loess samples gives a curve with a different slope (figure 3). However, the reference carbonate measurements, made by acid neutralization, are very questionable. DTA carbonate percentages (figure 2a, table I) agree well with X-ray analysis.

It may also be shown that DTA peak height is particularly sensitive to thermocouple alignment. An older calibration curve (figure 3) gives even a different slope.

In conclusion it is suggested that the DTA method may be used as an accurate means for carbonate determination if careful calibration tests are performed for each different type of soil (figure 2a). The next phase of work would be to study further effect of matrix, and investigate the reactions with dolomite[7].

X-RAY METHODS

Carbonates are determined by two methods of X-ray analysis. In one the sample is subjected to hard, short wavelength X-rays, and secondary fluorescence is measured[11]. This method measures total calcium and/or magnesium, but is not specific for carbonates. The second method is by X-ray diffraction and measurement of the intensity of the diffraction peaks. This

method is specific for calcite, dolomite, or other carbonate minerals. Both methods may be rapid, since no weighing or pretreatment is required.

Because of the drawbacks in chemical tests and DTA, a preliminary investigation was made to determine if one of the X-ray methods might be more accurate and practical. The first problem was to ascertain sensitivity and linearity of relationships. For this purpose the artificially prepared reference soils were used.

X-RAY FLUORESCENCE

Soil samples bombarded with hard X-rays fluoresce, or give off their own radiation with wavelengths and intensities characteristic of elements in the sample. To test for calcium, $CaK\alpha$ radiation is isolated for intensity measurement by diffraction at the Bragg 2θ angle from an EDDT crystal. The fluorescent radiation was counted with a gas flow counter tube. Each N value reported is the average of ten counts of ten seconds each. Use of an automatic calculating machine in conjunction with the counting allows computation of variance and 95% confidence intervals while the counts are being made[1].

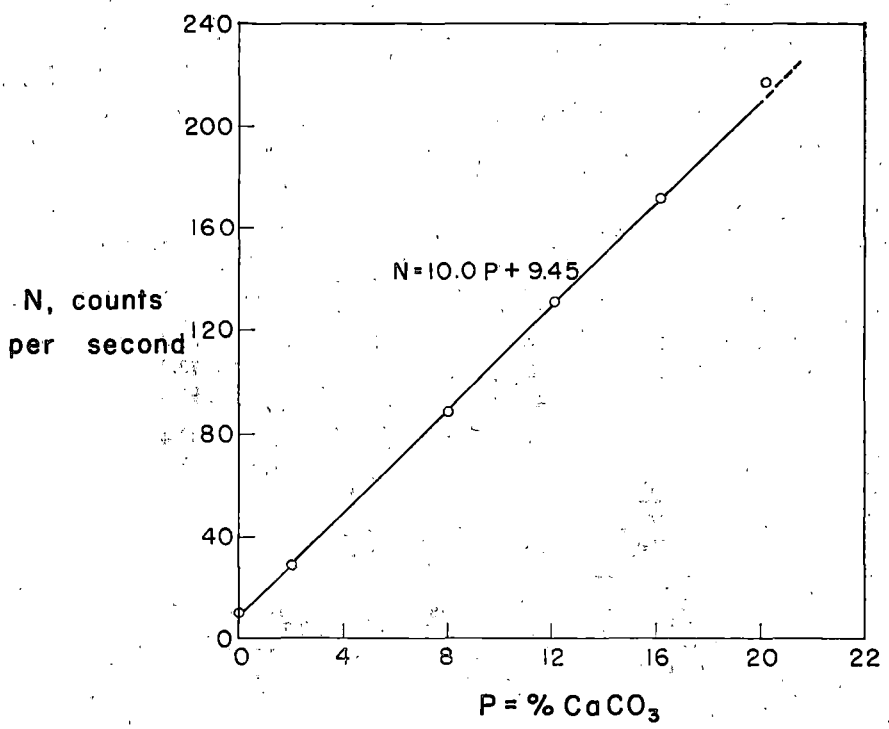

Fig. 4. X-ray fluorescent calibration curve for calcium carbonate. Since X-ray fluorescence measures total calcium, some counts result from samples with zero percent carbonate.

Samples were pulverized and packed dry into sample holders, and background counts were made on each sample at an angle of 2θ less 1°. Background counts were subtracted from the counts for $CaK\alpha$. A reference standard sample of powdered C. P. calcium carbonate was found to give an average $CaK\alpha$ count of 17,360 cps; if the count on this reference changes, results may be adjusted by multiplying by a proportionality factor.

The excellent linearity and low scattering of points suggests that X-ray fluorescence is an accurate method for carbonate determination (figure 4). Also it is very rapid, requiring about five to ten minutes per sample, including corrections for background and calculations for variance.

The major influence of matrix is to reabsorb fluorescent X-rays before they leave the sample, the amount of absorption depending on the other elements present. Therefore elements having high absorption coefficients will reduce X-ray intensities. Fortunately most of the common elements in soils have approximately the same absorption coefficients for $CaK\alpha$ radiation (table II). Chlorine, sulfur, and phosphorus are the critical elements with very high absorption coefficients, but these are not common in soils. Therefore the influence of changes in matrix may be small so long as the differences are within reasonable limits. The calibration curve probably applies to a wide variety of Midwestern loess and glacial soils (figure 4).

TABLE II. MASS ABSORPTION COEFFICIENTS OF DIFFERENT ELEMENTS FOR $CaK\alpha$ AND $CuK\alpha$ RADIATIONS

Element	Atomic number	μ/ρ $CaK\alpha^*$ $\lambda = 3.36$ A	$CuK\alpha\dagger$ $\lambda = 1.5418$ A
C	6	43	4.9
Al	13	417	48.5
Si	14	520[3]	60.3
P	15	610[3]	73.0
S	16	780	91.3
Cl	17	870	103.4
K	19	~200‡	143
Ca	20	~200‡	172
Mn	25	280‡	284
Fe	26	312	324

*Handbook of Chemistry and Physics, Chemical Rubber Publishing Co. 35th Ed., 1953.
†Internationale Tabellen zur Bestimmung von Kristallstrukuren, Vol. 2, 1935.
‡Interpolated on basis of atomic number.

Another problem, calcium not occurring as carbonate, is indicated by the count of 9.45 on the reference soil with zero percent carbonate (figure 4). To see if this count differs in different soils, several leached loess soils hav-

ing zero percent carbonate were tested. Unfortunately the zero count was found to vary from 9.1 to 23.5 in different samples (table I). Therefore the best method for correction may be to leach each sample with HCl, count for non-carbonate calcium, and subtract this from the result for the untreated soil. If the average correction is taken as 1.5% and subtracted from fluorescent percentages, the results will be close to those from DTA. The most accurate procedure, however, would be to determine corrections for each sample by leaching and re-running.

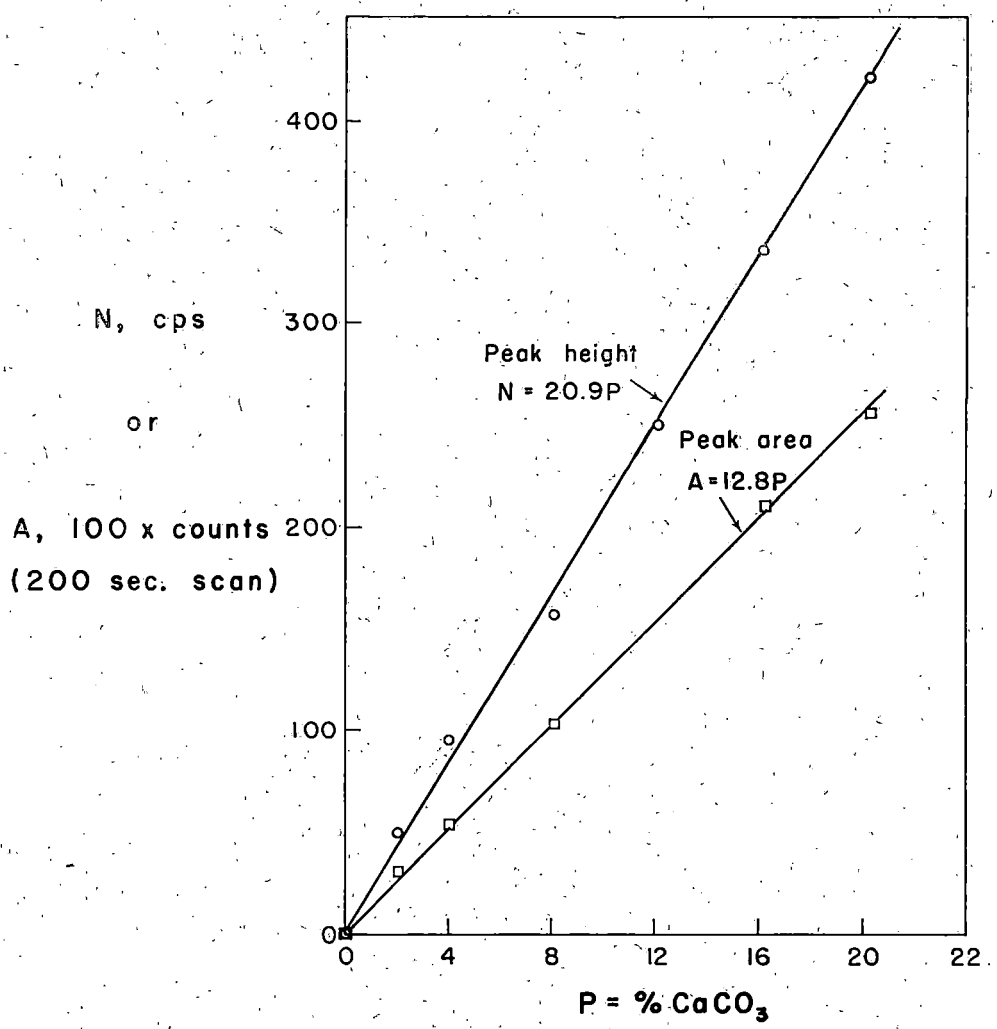

Fig. 5. X-ray diffraction calibration curves for calcite. Curves were fitted by least squares.

X-RAY DIFFRACTION

X-ray diffraction is a precise method for carbonate analysis, since it is specific for each crystal structure. In this study filtered copper $K\alpha$ radiation was used. Two counting methods were employed to measure the intensity of the diffraction: The peak height above background was obtained by first scanning with both increasing and decreasing 2θ angles to locate the center of the peak accurately, and the height was measured at the center of the peak by manual counting for 10 ten-second counts. Usually the 2θ angle was in the neighborhood of 29.2°, and background counts were made at a 2θ of 29.0°. In the other method the peak area was obtained by slow scanning (0.2° per minute) across the peak, usually 29.0° to 29.667°, and recording total counts, then subtracting counts for 29.0° background.

Results on artificially mixed soils indicate good linear response with no erroneous carbonate reported for the leached sample (figure 5). The scatter of points may be partly attributed to high background, but known percents carbonate of three out of the seven reference samples fall outside of the 95% confidence limits from counting, suggesting appreciable sample error (table III).

TABLE III. CARBONATE CONTENTS BY X-RAY DIFFRACTION PEAK HEIGHT METHOD

Sample	% $CaCO_3$	Counts per sec.* Peak	Background	N	% $CaCO_3$ from graph
Reference mixes					
R 0	0.00	118.8 ± 3.2	118.9 ± 1.8	0 ± 5.0	0 ± 0.25
R 83	2.03	165.6 ± 3.2	115.8 ± 2.5	49.8 ± 2.5	2.38 ± 0.27
R 35	4.05	215.7 ± 3.1	120.5 ± 3.2	95.2 ± 6.3	4.55 ± 0.30
R 65	8.10	278.5 ± 3.9	121.1 ± 2.6	157.4 ± 6.5	7.55 ± 0.31
R 1	12.15	362.6 ± 4.3	113.4 ± 2.9	249.2 ± 7.2	11.9 ± 0.3
R 19	16.19	454.3 ± 5.0	118.1 ± 2.7	336.2 ± 7.7	16.1 ± 0.4
R 51	20.24	545.4 ± 5.4	122.5 ± 3.8	422.9 ± 9.2	20.2 ± 0.4
Natural soils					
49-2	7.57†	135.3 ± 3.1	108.8 ± 3.0	26.5 ± 6.1	1.27 ± 0.29
23-1	12.3†	142.7 ± 3.2	112.2 ± 3.0	30.5 ± 6.2	1.46 ± 0.30
100-1	2.8†	138.0 ± 4.4	128.6 ± 2.3	9.4 ± 6.7	0.45 ± 0.32

*The ± entry indicates 95% confidence limits (0.754 times the standard deviation) for each value which is the average of 10 determinations.
†Versenate method for total acid-soluble Ca^{++} and Mg^{++}.

Scatter of points is less with the area counting method; the average error in seven determinations is 0.15% carbonate as against an average of 0.26% by the peak height method. The sample error appears to be reduced by scanning a diffraction peak rather than counting for maximum height. This suggests that the sample error may be from imperfectly random grain orientation, an effect much diminished by scanning. The scanning method is also the more rapid method for routine analysis.

As in the fluorescent analysis, the next question is absorption of X-rays by the samples, particularly when samples vary. Absorption of diffracted CuKα X-rays can vary considerably depending on the elements in the sample, the worst sponge being iron (table II).

TABLE IV. CARBONATE CONTENTS BY X-RAY DIFFRACTION PEAK AREA METHOD

Sample	% $CaCO_3$	Total Counts*			% $CaCO_3$ from graph
		Peak	Background	*A*	
Reference mixes					
R 0	0.00	24,000	23,780	220	0.17
R 83	2.03	24,910	21,900	3,010	2.35
R 35	4.05	29,450	24,100	5,350	4.14
R 65	8.10	34,520	24,220	10,300	8.05
R 1	12.15	38,240	22,680	15,560	12.2
R 19	16.19	44,910	23,620	21,290	16.6
R 51	20.24	50,430	24,500	25,930	20.2
Natural soils					
49-2	7.57†	25,150	22,230	2,920	2.28
		24,730	21,740	2,990	2.34
		24,210	21,250	2,960	2.31
23-1	12.3†	28,640	22,450	6,190	4.83
100-1	2.8†	25,770	24,660	1,110	0.866

*Peak scanned at 0.2° 2θ per minute from $2\theta = 29.0°$ to 29.667°. Background counted at $2\theta = 29.0°$.
†Versenate method for total acid soluble Ca^{++} and Mg^{++}.

Several loess samples were tested by X-ray diffraction (tables III, IV). Intensities were far below those from prepared samples, probably because acid leaching of the prepared mixes removes much of the iron. The count-scan method gave higher percentages than peak counting, indicating diffraction line broadening due either to poor crystallinity or fine crystallite size of the carbonate. The scan method appears to be the best one, but quantitative diffraction work will require use of internal standards or a different target X-ray tube.

CONCLUSIONS

1. CO_2 evolution methods are accurate but slow, and great care is required in the analysis. Errors result from adsorbed and dissolved gases.

2. Measurement of carbonates by acid neutralization is rapid but it is only approximate, due to other minerals which may react. In loess soils the error is commonly plus one or two percent carbonate, regardless of whether samples are calcareous or leached.

3. Versenate solutions may be used to distinguish calcium and magnesium ions in the acid leachate, but results are in error because of exchangeable calcium held on the clay. With loess the error is commonly plus 1 to 2.5 percent carbonate, regardless of whether samples are calcareous or leached.

4. Less serious error is introduced by reporting dolomite as calcite.

5. Differential thermal analysis is specific for calcite or dolomite. For best quantitative results DTA peak height is measured, the atmosphere during the run being air. DTA unfortunately is quite sensitive to changes in furnace and thermocouple alignment. One run requires about 1½ hours. Equipment maintenance costs are high.

6. Since X-ray fluorescence measures total calcium in a sample, the results are similar to those from versenate testing, except that magnesium is measured separately if at all. The X-ray method requires about five to ten minutes per sample compared with about one hour for versenate testing, and trace amounts of calcium may be detected. Correction for non-carbonate calcium may be made by acid leaching and re-testing. Absorption is not expected to vary appreciably in common Iowa soils, but can be corrected for by use of internal standards. Of the various test methods investigated, X-ray fluorescence appears most worthy of future investigation and use.

7. X-ray diffraction with CuKα radiation is accurate but overly sensitive to presence of iron, which absorbs the CuKα X-rays. Best results are obtained by count-scanning the diffraction peak and making allowances for background. Better results could be obtained with use of a different tube.

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CLAY FRACTION IN ENGINEERING SOILS: IDENTIFICATION BY DIFFERENTIAL THERMAL ANALYSIS

by

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INTRODUCTION

The clay fraction of soil is the seat of varied and vigorous reactions which greatly influence the behavior of soil as an engineering material. Experimental evidence indicates that such properties as plasticity, adsorption, shrinkage, swelling, and bonding strength are largely governed by the amount and nature of the clay material.

Many of the problems in soil engineering can best be attacked through a comprehensive study of the clay fraction and its relationship to soil behavior. Such a study will bring about a better understanding of the variables affecting soil behavior and will result in a more adequate definition and control of the properties of soil.

In the investigations of the clay fraction in soils the objectives were:

1. To specify tools and methods suitable for engineering laboratory use by which the clay fraction may be analysed.
2. To study the role of the clay fraction in soils to determine the variables involved and their influence on engineering properties.
3. To obtain a basis upon which the analytical results may be applied to determine and control the engineering behavior of soils.

The differential thermal method of analysis is a rapid, relatively accurate means for analyzing engineering soils qualitatively for certain constituents, particularly the clay minerals. In this method the procedures of sample preparation by which the sensitivity and usefulness of the method may be greatly increased are important, as are the interpretations of patterns obtained from these samples.

Structure of the Clay Minerals

To understand the properties and methods of analysis of the clay fraction, some knowledge of the chemical and structural makeup of the major clay minerals is essential. The predominant clay minerals in engineering soils may be classified in three groups, kaolinite, montmorillonite, and illite.

The basic structural unit of all the minerals in the three groups is the silicon-oxygen sheet having the formula $(Si_2O_5)^{-2}$. Each silicon ion is surrounded by four oxygen ions, and each group shares three of the four oxygen ions with its neighboring groups (figure 1). The valence requirements

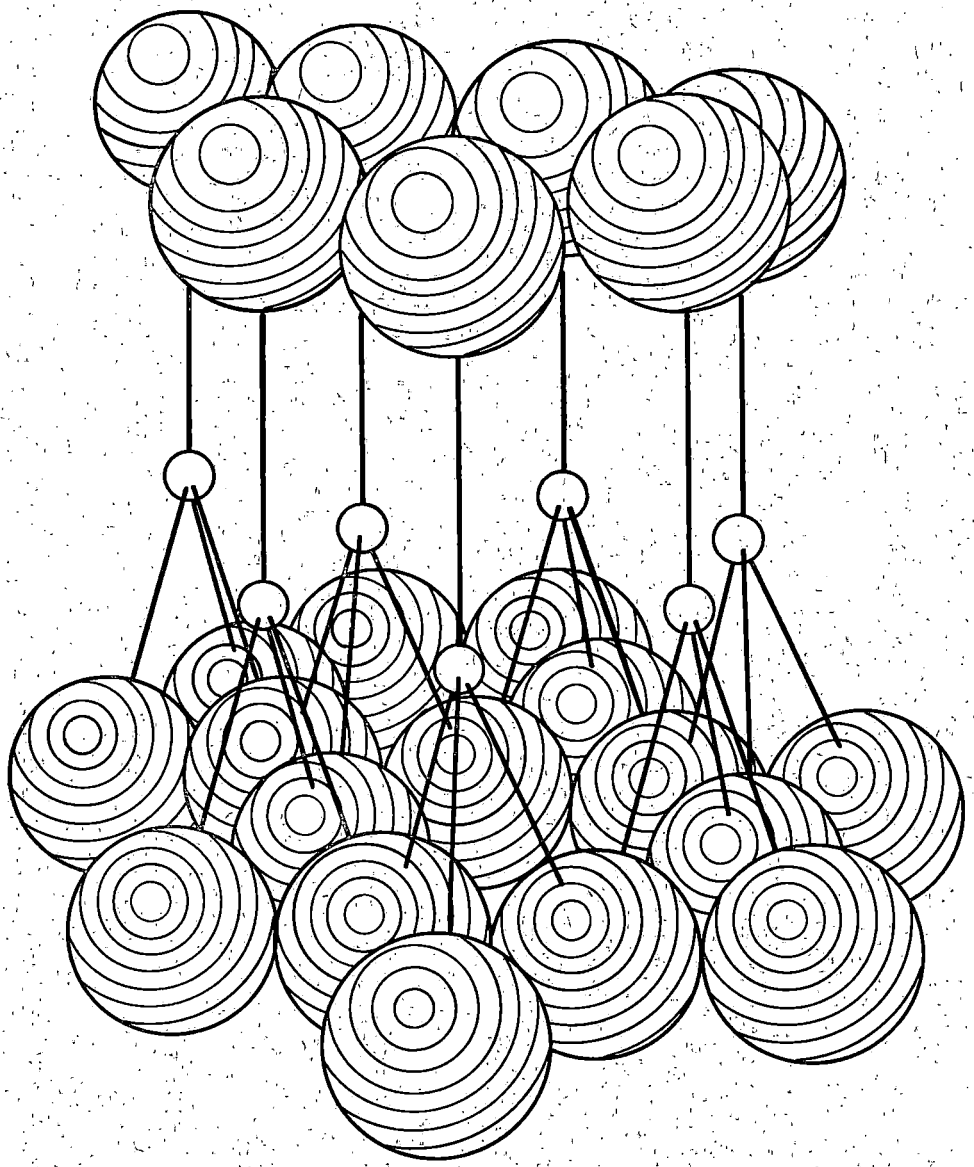

Fig. 1. View of $(Si_2O_5)^{-2}$ Sheet (Large spheres - oxygen ions)
(Small spheres - silicon ions)

of the oxygen ion directly above the silicon ion are unsatisfied. It is therefore available to other ions for bonding.

The only mineral of the kaolinite group in any abundance is the parent mineral kaolinite; macrite and dickite are scarce and differ from kaolinite only in minor structural features. All forms have the composition $(OH)_4Al_2Si_2O_5$. The structure of this mineral may be thought of as consisting of one basic silica sheet, with the aluminum ions bonded to the unsatisfied oxygens. Since each aluminum ion must be surrounded by six negative ions, hydroxyl ions $(OH)^-$ are necessary to complete the structure and to satisfy all the valence requirements.

The basic formula for the minerals of the montmorillonite group is $(OH)_2Al_2(Si_2O_5)_2$. This may be considered as being composed of two silica $(Si_2O_5)^{-2}$ sheets joined together by aluminum ions, with the hydroxyl ions $(OH)^-$ again being present to complete the structure. However, the actual chemical compositions of the minerals of this group vary considerably, owing to the possibility of such ions as iron and magnesium acting with the aluminum ions and partially replacing them for the bonding together of the silicon-oxygen layers. The nature of the mineral is such that it may take up considerable water between the platy particles and thereby swell markedly.

The illite group of minerals has approximately the same basic formula as the montmorillonites. However, in this group some of the silicon ions in

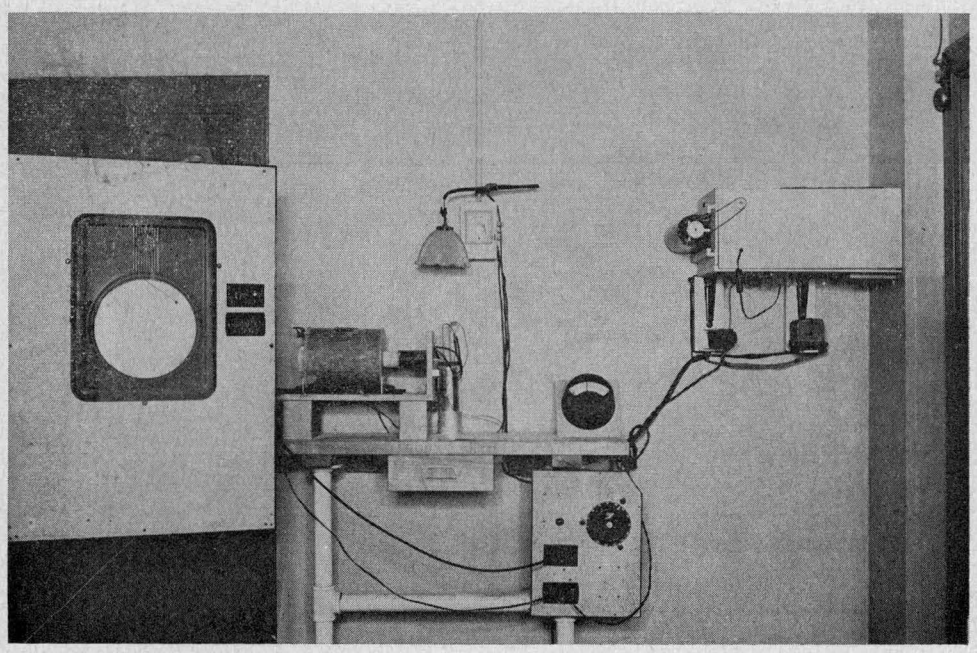

Fig. 2. Thermal analysis apparatus.

the basic sheet have been replaced by aluminum ions. This gives rise to a deficiency in charge which is made up by the inclusion of potassium ions between the particle layers. These potassium ions are actually bonded to adjacent particles and tend to restrict the movement of the sheets. The formula for this group may be expressed as $Ky(OH)_2\ Al_{2+y}\ Si_{4-y}\ 0_{10}$, the value of y varying from .5 to .75. Again, as with the montmorillonites, the aluminum ions are partially replaced by iron and magnesium ions. Water is also taken up between the particles, but swelling is limited by the presence of the potassium ions between the particles.

Differential Thermal Analysis

The differential thermal method of analysis has as its basis the heat effects accompanying the reactions which occur in a substance upon heating. When a material is heated, it may undergo one or more reactions which either give off heat (exothermic) or absorb heat (endothermic). A common example of an exothermic reaction is the burning of organic matter upon heating; an illustration of an endothermic reaction is the break-down of $CaCO_3$ into CaO (lime) and CO_2. Many minerals undergo characteristic reactions, such as loss of water, break-down of the crystal structure, mineral inversion, crystallization, etc., at known temperatures, and these reactions form the basis for their identification. The differential thermal analysis utilizes this fact by comparing the temperature of the sample with that of an inert standard while the two materials are being heated through a given temperature range, usually 50° - 1000°C. Since the temperature at which these reactions occur varies with the rate of heating, a constant rate, generally 10 - 12°C per minute, is used. This enables one to utilize standard thermal curves to interpret the results.

The apparatus employed to obtain the test curves consists of a furnace with adequate control to give the proper heating rate, a temperature measuring unit, and a means of determining the differential between the sample and standard. A number of workers have described various types of apparatus.

The apparatus used in this work was designed and built in the Ceramic Engineering laboratory. It is so constructed that it can be operated fully automatically or manually. The furnace is heated by Kanthal wire wound around an alumina tube. The temperature is controlled for automatic operation by a Bailey Pyrotron controller recorder; manual control is by a Variac in the furnace circuit. The temperature difference between sample and standard is determined by the use of a differential thermocouple, which is essentially two thermocouples joined so as to have opposing electromotive forces. The junctions of this couple are set in the middle of two holes in a nickel block. These holes serve as the holders for the sample and standard which are packed carefully around the thermocouple wires (figure 3). Any

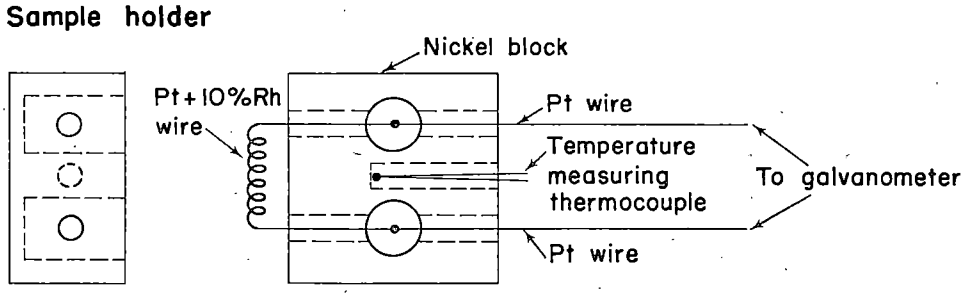

Fig. 3. Diagram of the sample holder.

temperature difference between the sample and standard gives a resulting o.m.f., the magnitude of which is proportional to the temperature difference and the direction of which is dependent upon the relative temperatures. The leads from the differential thermocouple are connected to a galvanometer through a variable resistance. The deflection of the galvanometer is recorded on photographic paper by reflecting a light beam from a mirror mounted on the galvanometer suspension, thereby recording the thermal curve. A means is also provided to mark the curve at 50°C intervals.

The sensitivity of the apparatus is controlled by the variable resistance in series with the galvanometer. Some reactions are accompanied by a relatively large heat effect and require only moderate sensitivity, whereas for others a very sensitive apparatus is needed for detection. The ultimate sensitivity is limited principally by the ambient effect of the furnace heating as reflected in the temperature at the center of the samples due to the difference in thermal conductivity of the sample and standard. This effect may be controlled somewhat by using as a standard sample material which has been subjected to previous heat treatment, and by packing both the sample and standard to approximately the same density. To compare the thermal curves obtained from different samples, a uniform amount of material should be used.

Sample Preparation

For analyses of relatively pure minerals or of a mixture which contains the mineral of interest as a major component, little or no sample preparation is needed. However, to investigate minor constituents, it may be necessary to so treat the sample as to increase the relative amount present or to remove those components whose heat effects may interfere with the desired reaction. The absolute percentage of a mineral which will give an interpretable reaction depends upon the amount of heat released or absorbed by

the reaction; and, therefore, that percentage is different for each substance, the actual minimum being determined only by experimentation.

Since the clay fraction in some engineering soils is in amounts too small to give a definite characteristic pattern, one of the major objectives of this work is to present a rapid method for increasing the concentration of the clay fraction and to illustrate how this method may be applied to certain soils. For this purpose seven soils were used. They are designated descriptively as: (1) Harrison County loess, (2) Johnson County loess, (3) Edina sub-soil, (4) Kansan gumbotil, (5) Maryland soil-aggregate, (6) synthetic soil, and (7) Webster topsoil.

The Harrison County loess is thought to be of eolian origin and was obtained from the deep Peorian loess area bordering the floodplain of the Missouri River in western Iowa. The sampling pit was near the bottom of a 60 foot road cut about 4 miles west of the town of Magnolia. This silty loam material had a light grayish-yellow color and classified A — 4(8) by the revised Public Roads classification system.

The Johnson County loess also of Peorian sub-age, was sampled about seven feet from the top of a road cut near Iowa City in eastern Iowa. The sample had a reddish-yellow color and a silty clay loam texture. It classified A — 7 — 6(10).

The sample of Edina subsoil was taken from a roadside pit near the town of Corydon in Wayne County, southern Iowa. This dull gray B horizon soil had a clay texture and classified A — 7 — 6(20). The parent material (C horizon) of the Edina series is moderately deep to shallow loess.

The name Kansan gumbotil has been applied to the highly weathered grayish material derived from an overlying Kansan glacial till. The sample used was obtained from a surface exposure near the town of Corydon, Iowa. It had a clay texture and classified A — 7 — 6(19).

The soil designated Maryland soil-aggregate was rust-red in color and was obtained from a small pit in Prince George's County, Maryland, about 4½ miles south of the District of Columbia line just off the road to Indian Head, Maryland. The soil was supplied by the Division of Physical Research of the Bureau of Public Roads. Texturally it classified as gravelly sandy loam and by the revised Public Roads system as A — 2 — 6.

In connection with previous work at the Iowa Engineering Experiment Station, a synthetic soil had been prepared. The non-clay portion of the soil was Ottawa sand and the clay fraction Florida kaolin. This sample was used to illustrate the typical thermal reactions of a kaolinitic type soil. It classified texturally as sandy loam and A — 2 — 4 by the revised Public Roads system.

The sample of Webster topsoil was taken from the A horizon of a corn field about two miles north of the City of Ames in Story County, central Iowa. It had high organic matter content and a silty clay loam texture. It

classified A — 7 — 6(13). The parent material of the Webster series is Wisconsin (Mankato sub-age) till.

Thermal patterns were made for the following fractions of each of the soils listed above: (1) the entire soil, (2) that portion which passed a 270 mesh sieve, and (3) the minus one micron fraction. For the samples indicated as (1) the soil was ground to pass a 40 mesh sieve. For the other two samples 100 grams of the soil to be tested was mixed with about 700 milliliters of distilled water, and sufficient sodium silicate* was added to disperse the clay. The dispersed soil is then passed through a 270 mesh sieve and the residue discarded. For the minus 270 mesh fraction, the suspension is acidified with a few drops of hydrochloric acid. This causes the clay to flocculate, or form large aggregates, and the solids settle rapidly. In this work, the rate of settling was accelerated by the use of a centrifuge, the entire settling taking only a few minutes.

For the finest fraction, the suspension obtained by sieving is allowed to settle, and the less than one micron sample is siphoned off. The length of time settled and the amount of suspension siphoned off may be determined from Stokes' Law, governing the velocity at which a spherical body falls through a liquid. This may be written as:

$$v = \frac{2r^2g\ (S_s - S_l)}{9_\eta} \tag{1}$$

where:

v = velocity of settling in cm/sec.
r = radius of the particle settling in cm.
g = acceleration of gravity, 980 cm/ sec.^{2}
S_s = specific gravity of the solid, for clays averaging about 2.5
S_l = specific gravity of the water, roughly equal to 1
η = viscosity of the water, equal to approximately .01 poises

The values given above are only approximations, accurate values for water may be determined from any chemistry or physics handbook and vary with temperature. However, these values are accurate enough to give an indication of the time interval involved. The distance settled (l) in an interval of time (t) for 1 micron diameter particles may be calculated as follows:

$$v = \frac{l}{t} = \frac{2 \times .00005^2 \times 980 \times (2.5 - 1)}{9 \times .01}$$

$$v = 8.175 \times 10^{-5}\ \text{cm/sec.} = .2943\ \text{cm/hr.} \tag{2}$$

From equation (2) it may be calculated that a 1 micron diameter particle would settle approximately 7 cm. in 24 hours. Since the long arm centrifuge

* The amount of Na_2Si0_3 used depends on the type and amount of the clay fraction and is most easily determined by visual inspection. A properly deflocculated suspension has a uniform, fine-grained appearance as contrasted to the coarse appearance and rapid settling of an undispersed system. This contrast is readily observed, especially if a few drops of acid are added to another sample for comparison. A few experiments of this type should enable a person inexperienced in this field to recognize the desired state.

employed by the authors in these experiments is capable of developing a centrifugal acceleration of 220 times gravity, its use enabled the settling of one micron particles a distance of 5 cm. in approximately 4½ minutes. This made it possible to siphon off the top 5 cm., add water and re-mix, and repeat until sufficient amount of sample has been obtained. This procedure greatly decreases the time necessary for sample preparation.

The suspension containing the minus 1 micron fraction which has been removed is acidified and allowed to settle as is done with the fraction which passes the 270 mesh sieve. After settling, most of the supernatant liquid is poured off, and the remaining water is removed by filtering in a Beuchner vacuum filter. The resulting filter cake is air-dried in an air blast. The reason for filtering and air-drying is to preserve the reaction which occurs in the illite and montmorillonite minerals between 100°C and 250°C. Oven drying, although at a temperature only slightly above 100°C, will remove the major portion of this reaction. An illustration of the difference in the types of curves obtained may be seen in figure 4, curves 2a and 2b. Curve 2a was obtained from an oven-dried sample, curve 2b from an air-dried sample.

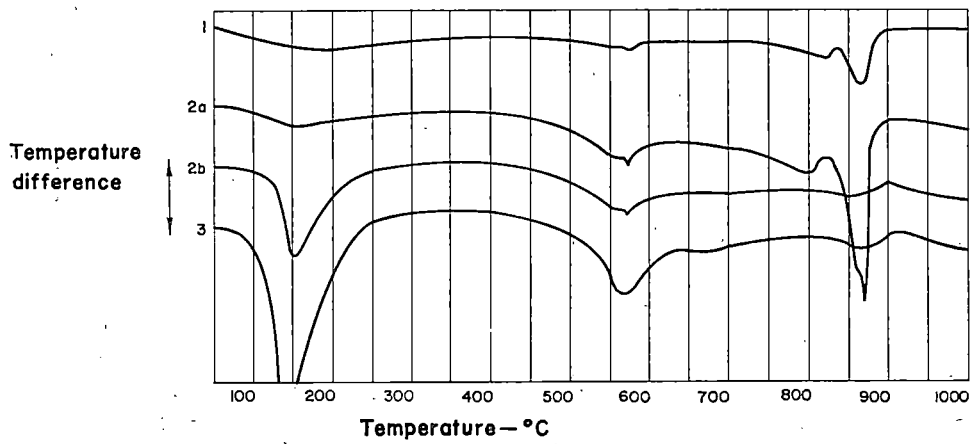

Fig. 4. Thermal curves of Harrison county Loess.

Sometimes, in addition to increasing the concentration of the clay fraction, it is necessary to remove constituents whose reactions tend to obscure the reactions which are desired. Organic material is particularly troublesome in this respect, since its reaction with oxygen causes large exothermic effects over almost the entire temperature range, and since it is so widespread in certain types of soils. Figure 4, curve 1, indicates the interference caused by organic material. Such material may be removed by treating the sample with hydrogen peroxide until no further reaction occurs. Cautious heating may be employed to hasten the reaction, but care should be taken

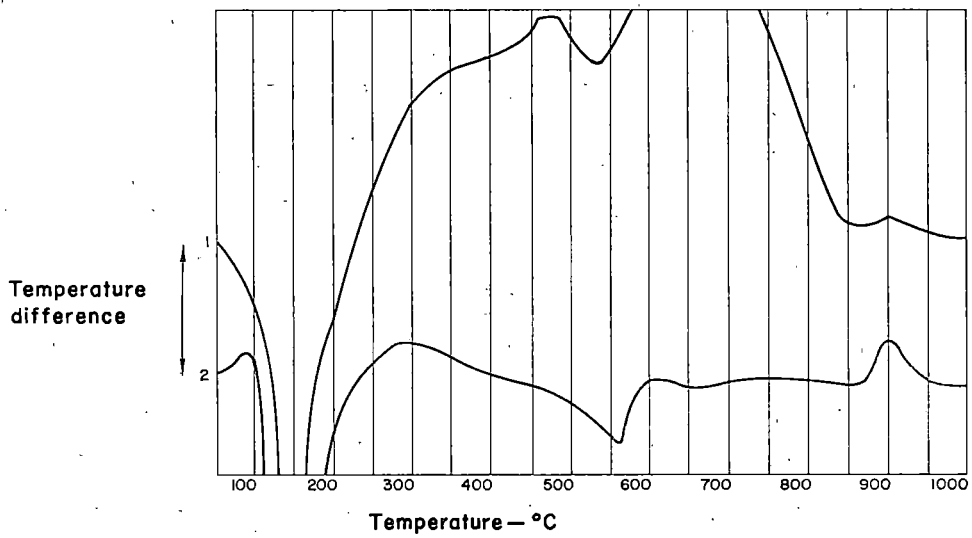

Fig. 5. Thermal curves of Webster topsoil.

that the evolved CO_2 does not cause the sample to froth over the container. The amount of H_2O_2 necessary depends on the sample and often the removal of the organic material is time consuming and laborious. However, when it has been completed, excellent patterns may be obtained on the sample, as is shown by figure 5, curve 2. Although a small amount of certain types of organic matter may be removed in the preparation of the less than one micron sample as shown in figure 6, this did not suffice for the organic content of Webster topsoil.

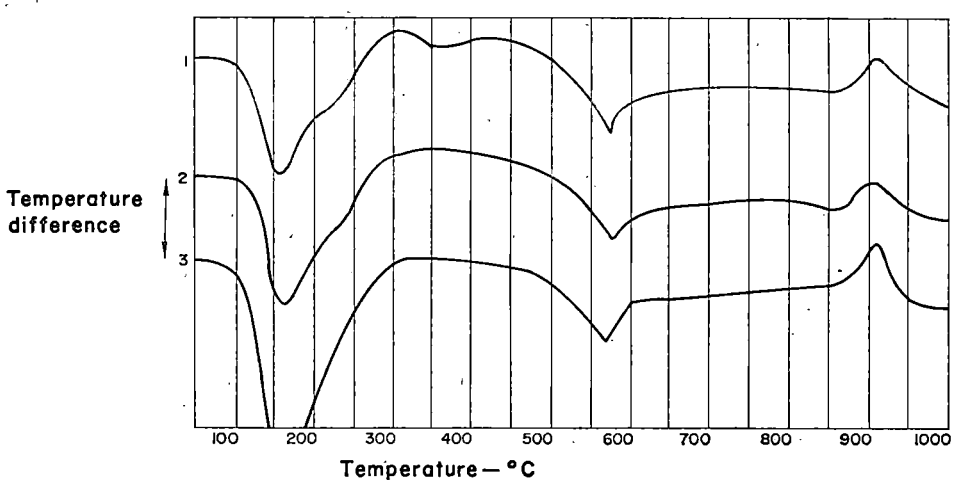

Fig. 6. Thermal curves of Edina subsoil.

Another group of interfering minerals which may be present are the carbonates, which give endothermic reactions between 750° and 900°C. However, these may be easily removed by the addition of a few drops of hydrochloric acid. In curves 1 and 2a of figure 4 is shown the typical carbonate reaction. This reaction, however, is absent from curves 2b and 3, since the sample preparation included the addition of acid. If the presence of the carbonates, limestone, dolomite, etc., is of importance, as it usually is in engineering soil studies, a pattern should be taken on untreated material as well as the acid-treated.

Interpretation of Analytical Results

Interpretations of the thermal curves obtained are based upon the pure mineral curves presented by Grim and Rowland[13]. Since the pattern for kaolinite is very distinctive, the curve obtained from the synthetic soil (quartz plus Florida kaolin) will serve to illustrate the characteristic reactions of this mineral. This is given in figure 7, curve 3, and shows a strong

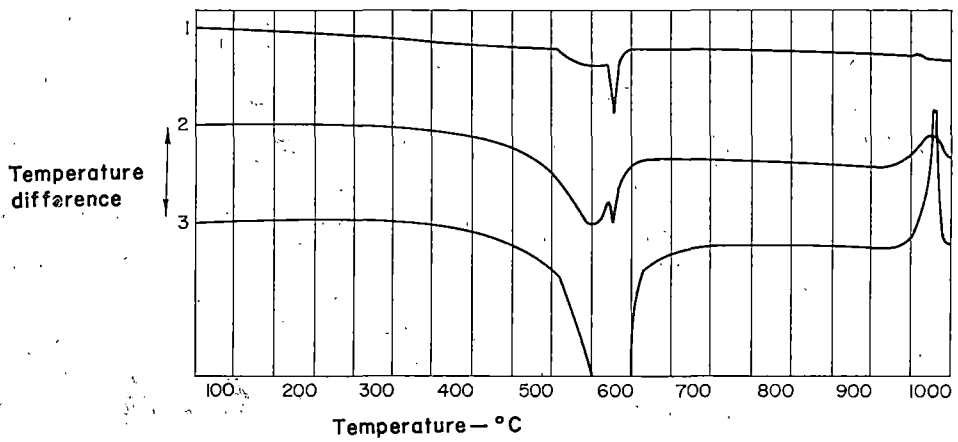

Fig. 7. Thermal curves of synthetic soil (sand and kaolin).

endothermic reaction centered between 550° and 600°C and a sharp exothermic peak at 980°C. However, since the patterns obtained from montmorillonite and illite are quite similar, let us examine Figures 5 and 6 which show the patterns obtained by Grim and Rowland on pure samples, the mineralogical contents of which have been determined by X-ray methods. It will be noted that both minerals give three endothermic and one exothermic reaction. However, only the second endothermic reaction may be used as a basis for differentiation. This occurs between 500° and 600°C in the case of the illites, between 600° and 700°C for the montmorillonites. An inspection of the curves of Figures 5 and 6 will show that this is true for each sample presented. It is upon these curves that the authors have based their interpretations of the soil analyses to be presented.

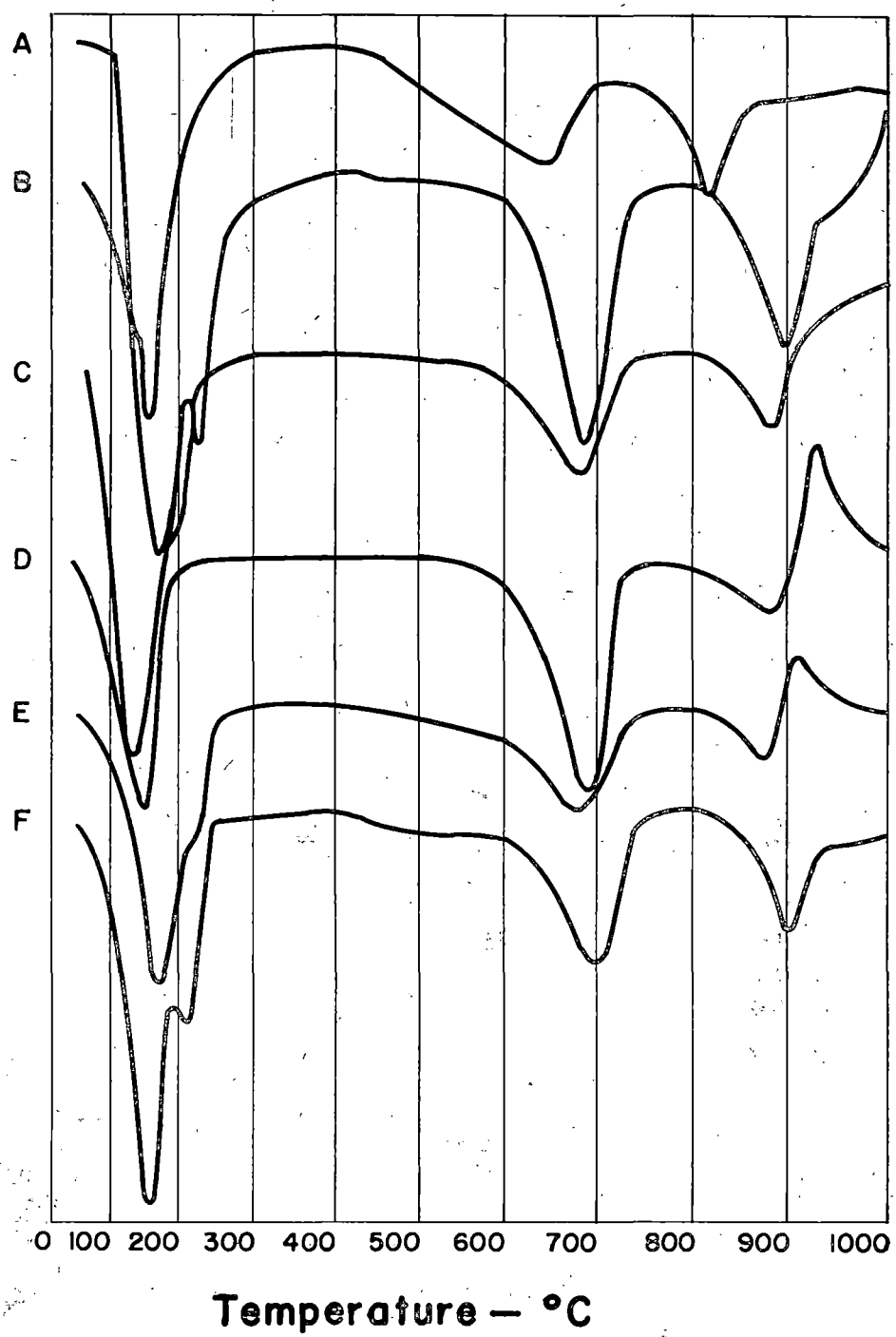

Figure 8. Thermal curves of samples of montmorillonite[6].

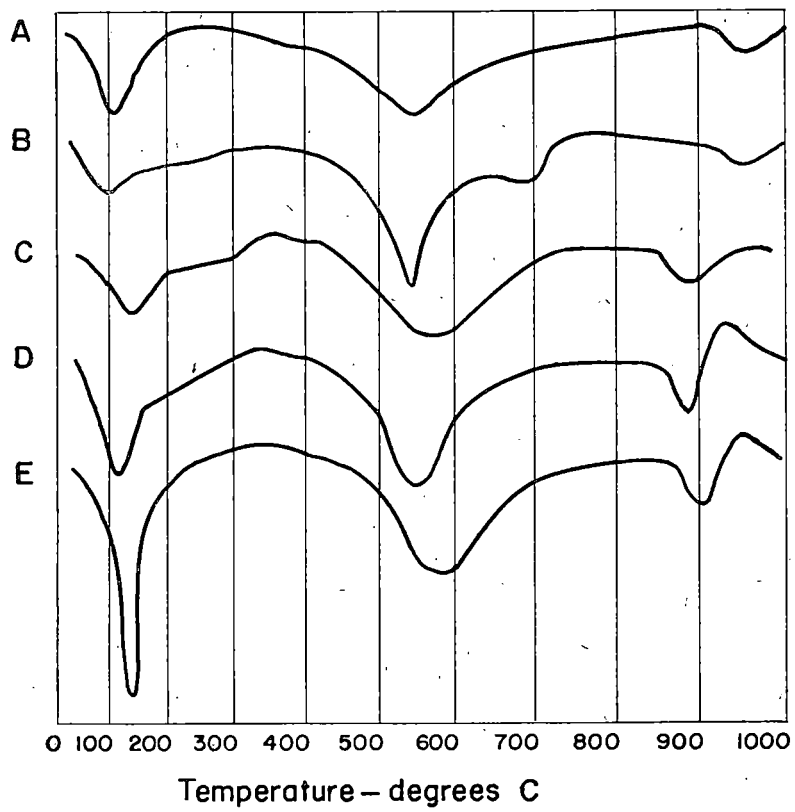

Fig. 9. Thermal curves of samples of illite[6].

Figures 4, 6, 7, 10, 11, and 12 present the patterns of the various samples studied. In each figure the individual curves are numbered 1, 2, and 3; these designate the original, through 270 mesh, and less than one micron samples respectively.

Figure 4 contains the analyses of Harrison County loess. Curve 1 shows the presence of quartz (endothermic at 573°C) and carbonates (double endothermic reaction, 800° - 900°C). The curves 2a and 2b were both made from the through 270 mesh sample; however, curve 2b shows the analysis of an acid treated, air-dried sample, and curve 2a is of a heat-dried sample. Although heat drying preserved the carbonate reaction, the loss of the low temperature endothermic reaction greatly reduced the value of the pattern for clay mineral determination. Curve 2b presents the typical illite pattern, and this is further confirmed by curve 3. It will be noted that the major portion of the quartz present in the soil was above one micron, since the quartz reaction does not appear in curve 3.

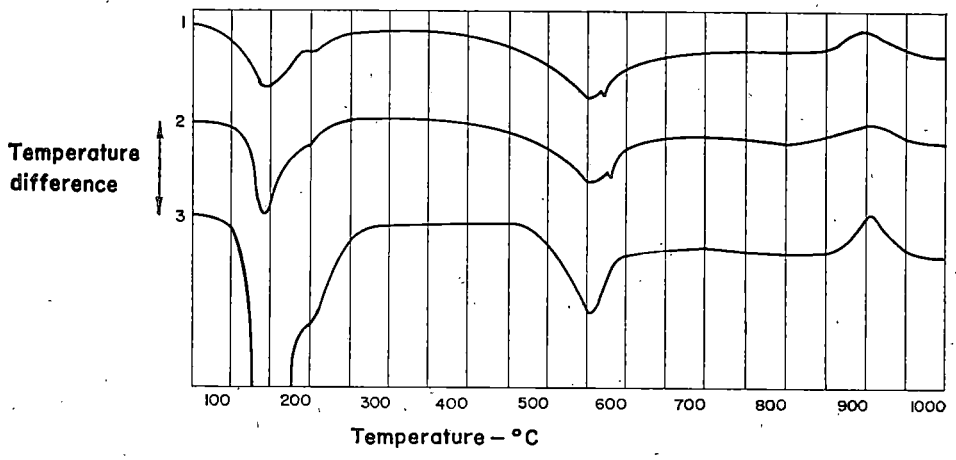

Fig. 10. Thermal curves of Johnson county loess.

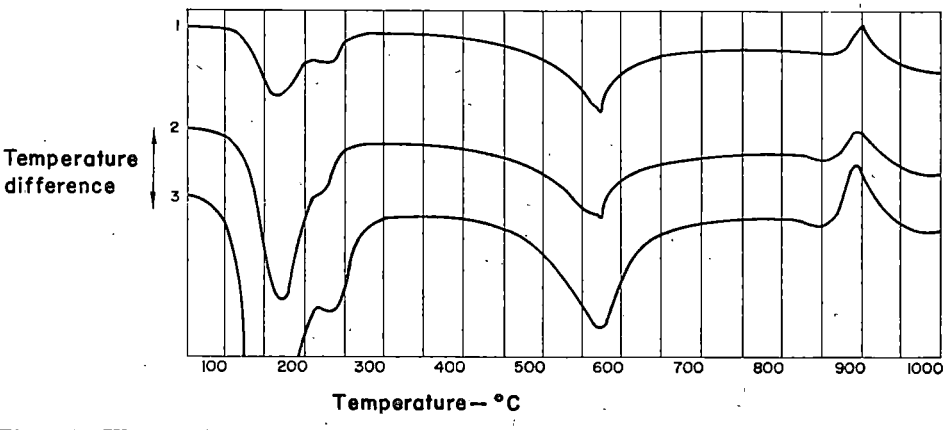

Fig. 11. Thermal curves of Kansan gumbotil.

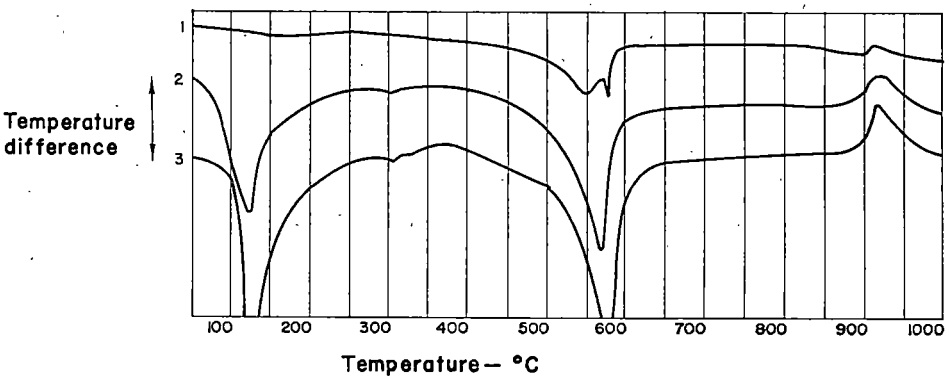

Fig. 12. Thermal curves of Maryland soil aggregate.

The Johnson County loess (figure 10) gives a typical illite reaction on all three samples. The curves also indicate that most of the sample is below 270 mesh size, since there is little increase in reaction magnitude between curves 1 and 2. This soil also contains quartz, as is readily evident by the 573°C reaction (high-low quartz transformation).

Figure 6 contains the thermal patterns of the Edina subsoil. This soil contains a large amount of clay fraction which is predominately illite. The broad exothermic reaction from 250° to 550°C is caused by the presence of some organic material. This reaction tends to partially mask the illite endothermic reaction at 550° - 600°C. No good evidence for the presence of quartz is indicated, since the sharp point at about 575°C may be caused by the combination of the reactions of the organic material and the illite.

Kansan gumbotil offers a series of interesting patterns, as given in figure 11. The presence of quartz is observable in curves 1 and 2. An examination of the standards of Grim and Rowland, figures 8 and 9, shows that the only samples exhibiting a double initial endothermic reaction are included in figure 8, that of the montmorillonites. No explanation of this behavior has been given by Grim or other workers; and, since it does not occur in all montmorillonite samples, it is the opinion of the authors that the position of the second endothermic peak, between 500° and 600°C, should constitute the major basis for analysis. On these grounds, the clay fraction must be classified as an illite.

Figure 12, the Maryland soil-aggregate sample, offers an example of a soil with a very large amount of coarse quartz and very little clay. However, comparison of curves 1 and 2 indicates that the major portion of the non-clay fraction is above the 270 mesh sample, curve 2.

As stated, the patterns shown in figure 7 were obtained from samples of a synthetic soil composed of sand and kaolin. The presence of the quartz sand is readily evident from the strong, sharp endothermic reaction at 573°C in curves 1 and 2. Curve 1 shows only a trace of the exothermic reaction at 980°C which is distinctive of the mineral kaolinite. It is interesting to note that the amount of clay in this soil was approximately five percent; therefore this seems to be the minimum amount which could be identified by this method. The kaolinite reactions become more pronounced in curve 2, and curve 3 shows clearly the characteristic reaction at 980°C.

A close examination of the patterns for the Harrison County loess (figure 4) and Edina subsoil (figure 6) reveals that a slight tendency toward an endothermic reaction exists between 650° and 700°C. This might be interpreted as being caused by a small amount of montmorillonite. Figure 13 presents the patterns obtained by Grim and Rowland[13] on prepared mixtures of illite and montmorillonite. A comparison between the figures 4 and

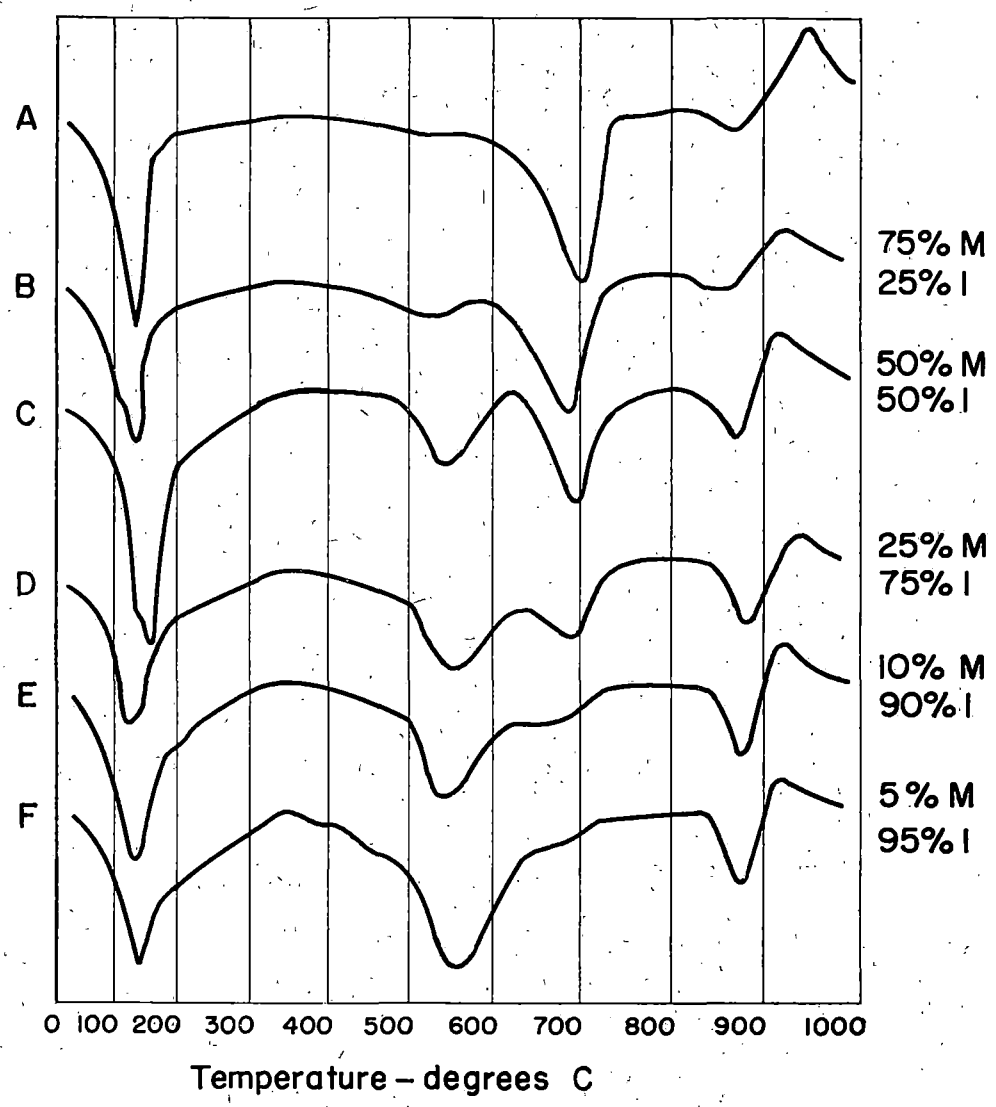

Fig. 13. Thermal curves of prepared mixtures of illite and montmorillonite[6].

6 and figure 13 indicates that, if it is present, montmorillonite occurs as less than five percent of the minus one micron fraction. Assuming a clay content of twenty percent, the amount of montmorillonite present would be less than one percent of the total soil. Similarly, since approximately five percent is the observable minimum for the mineral kaolinite and no typical 980°C exothermic reaction was observed on the soil studied, it may

be stated that if kaolinite is present, it occurs in amounts less than one percent of the total soil.

An interesting comparison may be made between the thermal patterns obtained on the original samples and the revised Public Roads classification. All soils having a classification of A - 7 - 6, denoting high plasticity, gave an interpretable pattern without treatment. These soils were Johnson County loess (figure 10), Edina subsoil (figure 6), and Kansan gumbotil (figure 11). The remaining soils, Maryland soil-aggregate (figure 12), Harrison County loess (figure 4), and the synthetic soil (figure 7), all required preparation of the soil to increase the clay concentration, and all have a low plasticity classification.

CONCLUSIONS

1. The mineralogical analysis of the clay fraction of soils by the differential thermal method may be greatly aided by proper preparation of the sample.

2. For some soils, containing small amounts of the clay minerals, identification by this method is not possible unless the soil is treated in such a manner as to increase the clay concentration in the sample.

3. Some materials, such as organic matter, whose reactions are objectionable in that they obscure those of the minerals for which the soil is being analyzed, may be removed by the proper soil treatment.

4. The difference in the extent and intensity of the thermal reactions obtained on the prepared samples gives a qualitative estimation of the particle size distribution in the soil.

5. The differential thermal method of analysis in conjunction with the proper soil preparation methods is a valuable tool for the analysis of the mineralogical content of soils and especially for the analysis of the basic clay content. However, the data obtained on the soil by this method constitutes only a part of that necessary for the complete analysis. Therefore its value is greatly increased by such complementary data as accurate particle size distribution and cation exchange capacity.

At the present time, perhaps the greatest lack in the data obtainable on soil samples, and on the clay fraction in particular, is in a clear, concise application of this data to the determination and control of the engineering properties of the soil. It is toward this end that the authors have begun the series of studies of which this is the first.

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CLAY FRACTION IN ENGINEERING SOILS: PARTICLE SIZE DISTRIBUTION AND CATION EXCHANGE CAPACITY

by

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(Highway Research Board Proceedings, 30:458-464. 1950.)

INTRODUCTION

Since the clay minerals in a soil occur for the most part in the colloidal fraction of engineering soils, any attempt to rationalize the properties of the soil must be directed not at the chemical nature but rather at those properties of the soil which predominate in the colloidal realm. As a basis for rationalization it is necessary that the distribution of particle sizes in the soil as a whole be known, including those particles in the minus one micron (0.001 mm.) size range. Similarly, since the exchangeable cations associated with the soil affect the colloidal behavior, a knowledge of the amount and type of exchangeable cations present is of great value.

Therefore, any work dealing with the study of the properties of the clay minerals must include a discussion of these variables both with respect to simple, accurate determinative methods and, of more importance, to a correlation of the data so obtained with some engineering properties of soils. A complete correlation is not possible due to the lack of sufficient data. However, a start may be made, and it is hoped that further studies will enable the soil engineer to utilize these tools more fully in the future.

Particle Size Determination

The measurement of particle size distribution in the colloidal range is a relatively recent development. The distribution of particles of microscopic size may be determined by counting techniques. But this is a tedious and time consuming procedure, and the information so obtained is not sufficient to warrant its use to any extent. Several methods for determining the size of fine particles have been proposed which are based on Stokes' Law for spheres falling through a medium. By means of this equation,

$$v = \frac{2r^2 g (S_s - S_l)}{9\eta} \qquad (1)$$

where:

v = velocity of settling
r = radius of the particle settling
g = acceleration of gravity
S_s = density of the solid
S_l = density of the suspending medium
η = viscosity of the suspending medium

it is possible to determine the rate of settling and from this the size of particles which have settled a given distance. The particle size distribution may then be found by determining the actual amount of material at any given depth at different times. The amount of material may be found by taking a sample at the desired depth and weighing the dried solids, as in the Andreasen pipette method, or by measuring the specific gravity of the suspension at that depth. Although the former method is the more accurate, it is time-consuming. For this reason, a variation of the latter method has been adopted for this work. The procedure is patterned after one proposed[2] in which the specific gravity of the suspension is measured by a soil hydrometer (range of 1.010 to 0.990).

In the measurement of particle size by settling rates, perhaps the most important single consideration is the dispersion of the solid into individual particles. Solids in the finer size range have a marked tendency to agglomerate unless the proper sample preparation is made. For this work the soil was dispersed by the addition of sodium silicate, the optimum amount determined by settling tests. A series of suspensions with varying amounts of electrolyte were made up and allowed to stand for several hours. The optimum amount of electrolyte was found by visual inspection of the suspensions, the best deflocculated having the smallest clear layer at the top and the least amount of solids settled out at the bottom. After the proper amount of electrolyte had been added, the soil sample being prepared for analysis was tumbled end over end for at least three days. The dispersion was then checked by microscopic examination.

The suspension employed contains 2 percent solids by weight, i.e., 2 grams of clay per 100 cc. of water. The specific gravity measured is assumed to be that of the suspension at the center of the hydrometer bulb; the particle size being investigated is that size which has settled from the surface of the liquid to the center of the hydrometer bulb. The hydrometer reading is used to determine the percentage of solids finer than that size by means of the following equation:

$$W \text{ percent} = \frac{S_s}{S_s - 1} \times \frac{100}{C} (S - S_l) \times 10^3 \qquad (2)$$

Where:
S_s = density of the solid
S = specific gravity of the suspension

S_l = specific gravity of the suspending medium
C = concentration of the solid, grams per liter.

Since the immersion depth of the hydrometer is variable, depending on the specific gravity, the calculation of equivalent spherical diameter of the particle is greatly simplified by the use of the Casagrande nomographic chart (obtainable from the Soil Mechanics Laboratory, Massachusetts Institute of Technology).

As may be seen from equation (1) the velocity of settling decreases as the square of the radius of the particle, i.e. a given particle will settle only one-fourth as rapidly as a particle twice its size. Therefore, it is desirable to increase the settling rate of the particles in the finer size ranges by the use of a centrifuge. In this way, the acceleration of the particles is increased over that obtained by gravity settling. In order to apply Stokes' Law to centrifugal settling it is necessary to substitute the acceleration obtained for the acceleration of gravity (g) in equation (1). This is calculated as follows:

$$\alpha = r\omega^2 \tag{3}$$

where:
α = acceleration
r = radius of circle of revolution
ω = angular velocity

The centrifuge used in this study is shown in figure 1 and is capable of developing an acceleration of 225 times gravity.

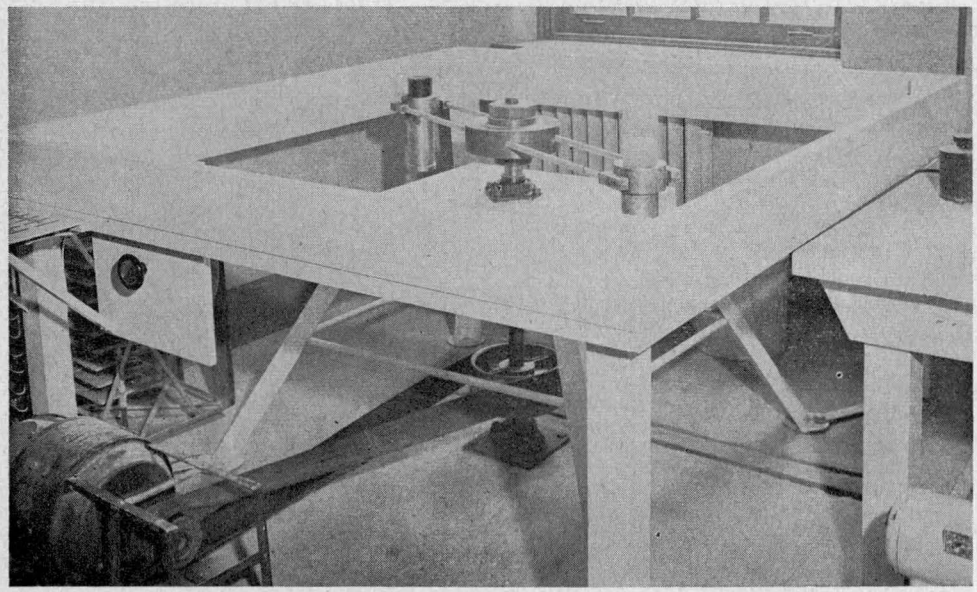

Fig. 1. Photograph of long-arm centrifuge.

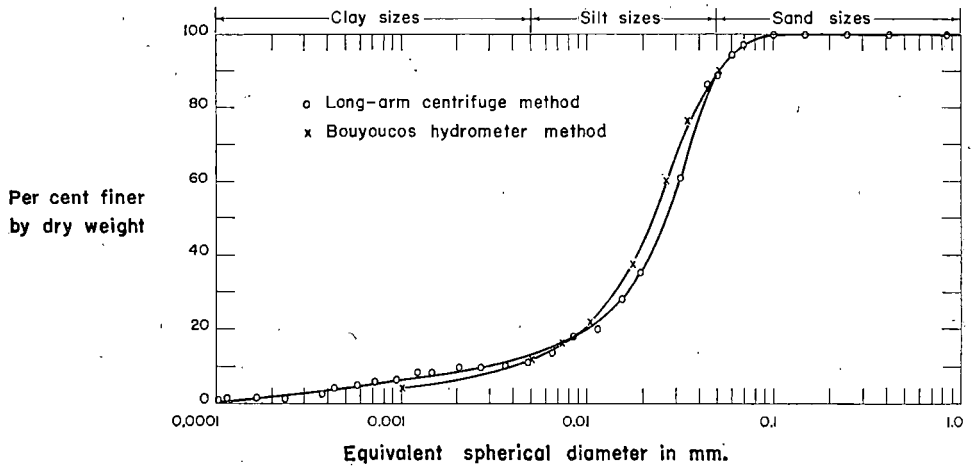

Fig. 2. Particle size distribution of Harrison County loess.

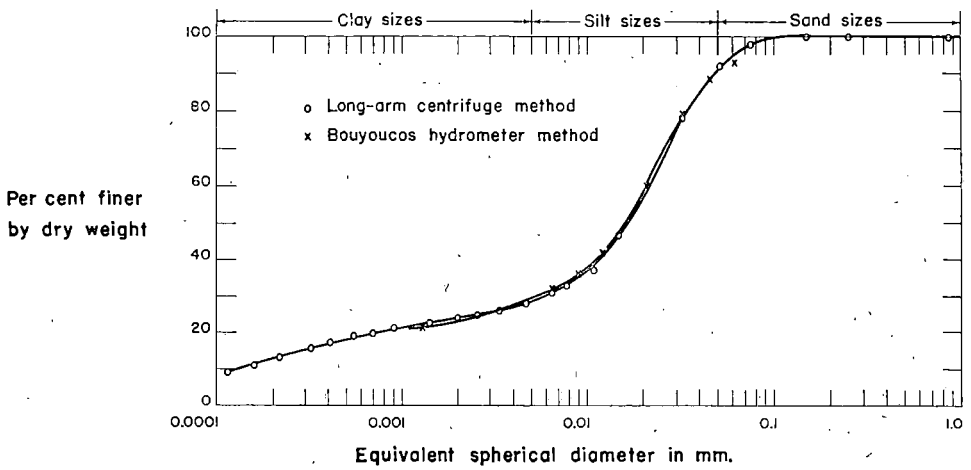

Fig. 3. Particle size distribution of Johnson County loess.

The data obtained from equations (1) and (2) are plotted on semi-log paper with the weight percent finer versus the logarithm of the particle size (figures 2, 3). The particle size distribution may be read directly from the graph as the weight percentage of soil particles whose diameters are less than any given size. The distribution may also be expressed in terms of the percent of the soil particles in a series of size ranges, e.g. clay (less than 5 microns), silt (5 microns to 50 microns), and sand (greater than 50 microns).

CATION EXCHANGE CAPACITY

A most important property of a clay mineral is its cation exchange capacity. This is closely connected to the physical behavior of the mineral, for example its plasticity and shrinkage. A knowledge of the cation exchange capacity of the mineral or soil is necessary for proper treatment of engineering soils, e.g. stabilization by organic cations.

The exchange capacity values vary widely depending on the type and particle size of the clay mineral. In general, it may be said that these values, expressed as milliequivalents of cation per 100 grams of soil are for:

Montmorillonite	85 to 100
Illite	25 to 60
Kaolinite	3 to 15

The clay minerals probably adsorb cations by one or both of two different mechanisms. Cations are attracted to the edges of the broken clay particle. It is known that the clay particles are mainly in the finest size range, and in this range the area of the broken edges becomes significantly large. Also as the size of the particles decrease, the edge area per unit mass increases. The atoms at the particle edge possess unsatisfied bonds, and it is by these unsatisfied bonds that the cations are attracted to the particle.

The second mechanism by means of which cations are held to the clay particle is the result of substitution of such ions as iron and magnesium for aluminum in the crystal lattice. Since ferrous iron and magnesium are both divalent, the substitution of these for trivalent aluminum gives rise to a deficiency of positive charge in the structure. This deficiency may be satisfied by cations which do not become an integral part of the lattice but are strongly attracted or adsorbed to the particles. As would be expected from the structures of the clay minerals, this second mechanism is operative only in the montmorillonites and illites; the adsorption of ions to the broken edges takes place both in these minerals and on kaolinite.

The adsorbed cations are attracted to the particle more or less strongly depending on the ion involved. The higher the charge on the cation the stronger is the attractive force, calcium being more tightly held than sodium. For ions of constant charge the force of attraction also varies and is believed by some to depend on the degree of hydration of the ion. The cation hydrogen in anomalous in its behavior and is more tightly held than any other cation.

When an electrolyte is added to a clay, the cation of the electrolyte tends to replace the cation adsorbed on the clay particle to a greater or lesser ex-

tent depending upon their relative valencies, their degree of hydration, their respective concentrations, and the degree of solubility of the chemical compound formed. For example, when a clay containing adsorbed calcium cations is treated with sodium chloride, the following chemical reaction occurs:

$$\text{Clay} - \text{Ca} + 2\text{NaCl} \rightleftarrows \text{Clay} - 2\text{Na} + \text{CaCl}_2 \qquad (4)$$

The equilibrium established will tend to favor the calcium clay since the calcium ion is more strongly attracted by the clay than the sodium and since (calcium chloride being soluble) no calcium ions will be removed from the solution. However if this same clay is treated with sodium silicate, the reaction will occur as follows:

$$\text{Clay} - \text{Ca} + \text{Na}_2\text{Si0}_3 \leftrightarrows \text{Clay} - 2\text{Na} + \text{CaSi0}_3 \qquad (5)$$

This reaction differs from that above in that one of the products is relatively insoluble, calcium silicate. Therefore any calcium ions replaced by sodium will be removed immediately from the solution in the form of calcium silicate and the reaction will progress to the right, forming a sodium clay.

The total amount of exchangeable cations associated with the soil available for exchange reactions, such as those described above, may be divided into two classes, the exchangeable bases such as sodium and calcium, and exchangeable hydrogen. This division arises from the fact that for most experimental methods for determining the amount of cation associated with a clay (the cation exchange capacity) these two quantities are determined separately. This is true for the method employed for this study. Numerous Methods for determining both base and hydrogen ion exchange capacity have been published. The method was selected because of its speed and because of the simplicity of the technique and of the equipment required[2]. The equipment needed for this determination is a pH meter capable of measuring pH to .02 pH units, neutral normal ammonium acetate, and 1 normal acetic acid.

To determine exchangeable hydrogen, 2.5 gm. of soil is placed in a 50 ml. Erlenmeyer flask, 25 ml. of neutral normal ammonium acetate is added, the flask is stoppered, and the 1 to 10 mixture is allowed to stand for 1 hour with occasional shaking. The pH is determined on the mixture. To determine total exchangeable bases, 2.5 gm. of soil and 25 ml. of normal acetic acid are mixed and treated in the same manner as the mixture prepared for exchangeable hydrogen determination. The pH of the mixture is determined.

The number of milliequivalents of exchangeable hydrogen and exchangeable bases are found from figures 4 and 5 respectively. Figure 4 was obtained by potentiometric titration of 100 ml. of neutral normal ammonium acetate buffer with 0.2N acetic acid. The number of milliequivalents resulting in a

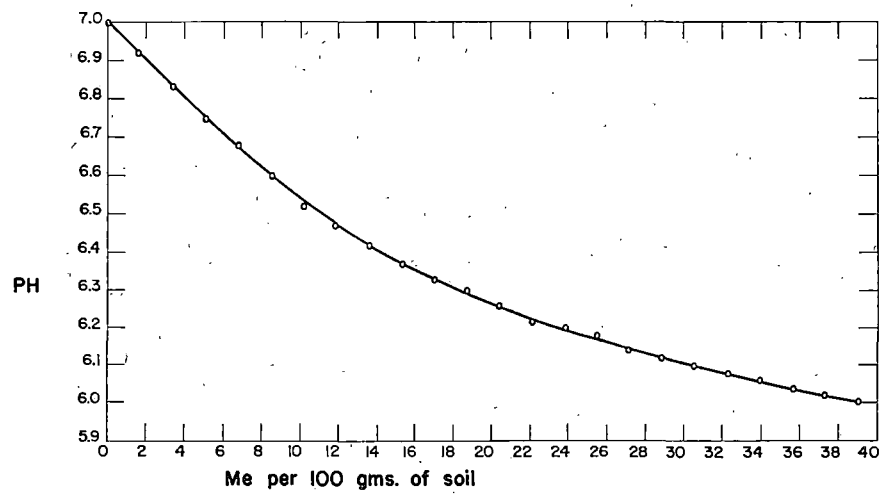

Fig. 4. Potentiometric titration of normal ammonium acetate.

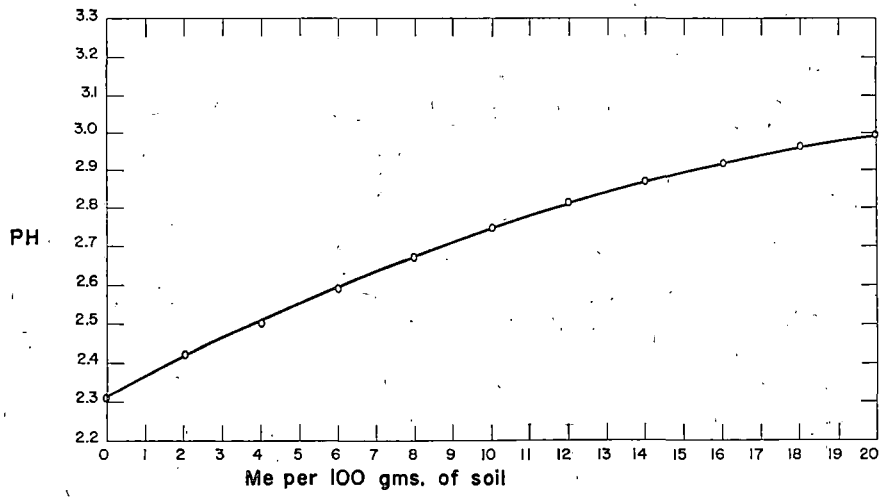

Fig. 5. Potentiometric titration of normal acetic acid.

given pH value is then multiplied by 10 so as to express the results in the units of milliequivalents per 100 gms. of clay, the usual manner of expressing exchange capacity. The factor of 10 is necessary, since a soil-buffer ratio of 1 to 10 as selected by Brown would require 1000 ml. of buffer solution for 100 gms. of soil. Since only 100 ml. of buffer solution is employed for the standard curves, the results must be increased tenfold to give the total milliequivalents for 1000 ml. of buffer. Figure 5 is obtained in a like

manner by titrating normal acetic acid with 0.2 normal ammonium hydroxide.

This method was employed to determine the exchangeable hydrogen and exchangeable bases on samples of two Iowa soils: Harrison County loess and Johnson County loess. The Harrison County loess is thought to be of eolian origin obtained from the deep Peorian loess area bordering the floodplain of the Missouri River in western Iowa. The sampling pit was near the bottom of a 60 foot road cut about 4 miles west of the town of Magnolia. This silty loam material had a light grayish-yellow color and classified A - 4(8) by the revised Public Roads classification system. The Johnson County loess also of Peorian sub-age, was sampled about seven feet from the top of a road cut near Iowa City in eastern Iowa. The sample had a reddish-yellow color and a silty clay loam texture. It classified A - 7 - 6(10).

Cation exchange capacities were also determined for the minus 1 micron fraction. To decrease the time necessary for settling, the samples were treated with hydrochloric acid and then washed with distilled water to remove the excess acid, the hydrogen ions partially replacing the bases on the soil in the process (table I). The results obtained on the same soils using the calcium oxalate method are also in the data[3].

TABLE I. CATION EXCHANGE CAPACITIES, M.E./100 GMS.

Soil Sample	Exchangeable Bases	Exchangeable Hydrogen	Total Exchangeable Cations	Calcium Oxalate Method[3]
Johnson County loess whole soil —	8.8	3.7	12.5	14.1
—1 micron fraction	2.8	42.5 43.4*	45.3 46.2	45 – 50
Harrison County loess whole soil	1.8	9.8	11.6	11.5
—1 micron fraction	4.0	43.0*	47.0	45 – 50

*Values determined using a ration of 1 to 20, soil to buffer.

RESULTS

The results obtained from the particle size distribution determination indicate that the Johnson County loess contains a much larger amount of finer material than the Harrison County loess. The lower limit of particle sizes for the latter soil occurs at one-tenth micron; the particle sizes in the former soil extend below this point.

The method for determining cation exchange capacity was modified for certain phases of this investigation[2]. The standard titration curves given in the original article covered only the range 0 to 15 milliequivalents per 100 grams of soil; similar standard curves were prepared extending to 60 milliequivalents. The curve showing the titration of neutral normal ammonium

acetate with approximately .2 normal acetic acid, shows that, because of the changing slope, the useful range is limited to less than 20 milliequivalents (figure 4). Of course this is an arbitrary limit which is set only by the degree of accuracy desired for the result. To illustrate this: assuming an accuracy of .02 in the pH measurements, the degree of error is 0.4 m.e. at 2 m.e. At 35 m.e. the degree of the error is approximately 2 m.e. A portion of this error occurring in testing soil having a high exchange capacity may be eliminated by using a different ratio of soil to buffer. Since the curve is based on a ratio of 1 to 10, the results obtained on a mixture of 1 part of soil per 20 parts of buffer, when multiplied by 2, would give the capacity in terms of milliequivalents per hundred grams of soil. Experimental proof of this relationship is shown in the values obtained on Johnson County loess, a value of 42.5 m.e. resulting from a mixture of 1 part of soil to 10 parts ammonium acetate and a value of 43.4 being obtained by doubling the results obtained with a 1 to 20 mixture. Therefore the data on the titration of 1 normal acetic acid with .2 normal ammonium hydroxide, cover only the range of 0 to 20 milliequivalents (figure 5). Values exceeding 20 m.e. may be obtained by the above method.

A limitation of the cation exchange capacity method presented is its failure in soils such as Harrison County loess with a high percentage of carbonate minerals. The initial results determined on this soil by the usual procedure were obviously in error, since the pH of the ammonium acetate leachate actually increased slightly and the determination on the acetic acid leachate gave a result exceeding 100 milliequivalents. Therefore the soil was treated with dilute hydrochloric acid to remove the carbonates and then washed with distilled water in a vacuum filter to remove the excess acid. After acid treatment, the total exchange capacity as determined by the method presented agreed closely with that obtained by P. K. Fung (table I). As would be expected, this converted the major portion of the clay to a hydrogen clay, as may be seen by comparing the exchangeable hydrogen and exchangeable base values (table I). The determination on

TABLE II. PROPERTIES OF TWO LOESS SAMPLES

Soil properties	Harrison County Loess	Johnson County Loess
Liquid limit, %	31.9	33.8
Plastic limit, %	26.4	21.3
Plasticity index, %	5.5	12.5
Shrinkage limit, %	21.7	14.8
Shrinkage ratio	1.61	1.83
Volumetric change, %	26.7	52.5
Centrifuge moisture equivalent, %	16.6	20.9
Field moisture equivalent, %	23.7	31.5
Public Roads classification	A-4(8)	A-7-6(10)

the minus 1 micron samples also showed a high amount of exchangeable hydrogen because of its acid treatment during settling. It may be noted here that the differential thermal analysis method is an excellent method for the detection of the presence of carbonates.

Certain properties of the two loess soils are indicative of engineering behavior (table II). Comparison of this data with figures 2 and 3, showing particle size distributions, indicates that the soil containing the higher percentage of particles in the five sizes exhibits higher plasticity values, greater shrinkage, and a greater affinity for water, as would be expected. Correlation with cation exchange values cannot be made at this time due to a lack of fundamental research in this field. Another factor undoubtedly influencing these properties is the presence of carbonate minerals. Again sufficient knowledge is not available to evaluate accurately their influence.

CONCLUSIONS

Perhaps the most important conclusion that may be derived from the study presented above is that relatively simple, accurate methods for the determination of particle size distribution and cation exchange capacity are now available to the soil engineer; however, correlation of the results obtained by these methods with soil properties cannot be made at present because of the lack of basic knowledge in the fields of soil technology and engineering. Further studies with the purpose of making available the knowledge necessary for the fullest application of analytical research to practical engineering are in progress.

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CLAY FRACTION IN ENGINEERING SOILS: INFLUENCE OF AMOUNT ON PROPERTIES

by

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(Highway Research Board Proceedings, 31:558-563. 1952.)

ABSTRACT

Part III of the series describes the results of a study of one of the important variables affecting engineering properties of soil: the amount of clay present in the soil. Test data for the deep Wisconsin (Peorian) loess of southwestern Iowa affords a rare opportunity for such a study with natural soil. Graphs show that the liquid limit, plastic limit, plasticity index, shrinkage limit, centrifuge moisture equivalent, hygroscopic moisture, field (in-place) density, and field moisture content have an apparent linear relationship with 0.002-mm.-clay content. No simple relationship was found between the field-moisture equivalent and clay content.

Parts I and II of this series reporting on investigations of the clay fraction in engineering soils, give tools and methods suitable for engineering-laboratory use by which certain properties of the clay fraction may be analysed. One of the objectives of the investigations is to study the role of the clay fraction in soils to determine the variables involved and their influence on engineering properties, which are those physical properties of soils used in the design and construction of engineering works. This reports the results of a study of one of the important variables, the amount of clay present in the soil.

Test data for the deep Wisconsin (Peorian) loess in southwestern Iowa afforded a rare opportunity for such a study with natural soils.

Wisconsin Loess of Southwestern Iowa

Four glacial drifts of the Wisconsin stage, Iowan, Tazewell, Cary, and Mankato, have recently been mapped in Northwestern Iowa[5]. Each of these four glaciations is believed to have contributed to the formation of the composite Wisconsin loess (also called Peorian loess in the geological literature) which forms a massive surface deposit that mantles older loesses and pre-Wisconsin glacial deposits in southwestern Iowa. Evidence indicates that the main body of the loess is wind-blown material. Major sources of supply

were perhaps the flood plains of valleys draining the drift areas and also the raw surfaces of the newly deposited drifts.

The portion of the Wisconsin loess area being studied by the Iowa Engineering Experiment Station is shown in figure 1. The thicknesses of the

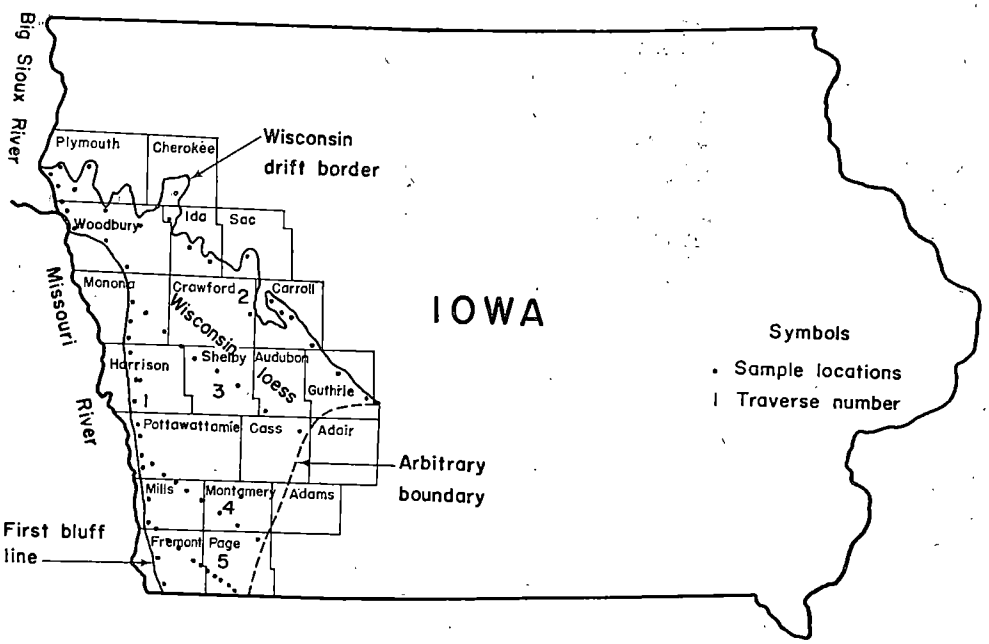

Fig. 1. Locations of sampling traverses in deep Wisconsin (Peorian) loess area of southwestern Iowa.

Wisconsin loess in this area have been mapped as greater than 17 ft. on ridges and hilltops[12]. Along the west boundary, the east valley wall or first bluff line of the Missouri River, the loess thicknesses are much greater, depth measurements of from 60 to over 100 ft. having been made[8, 9]. The loess thicknesses are also greater than 17 ft. on hilltops along the north boundary, the Wisconsin (or Iowan) drift border[14]. The depth of the Wisconsin loess becomes thinner in a southeasterly direction away from the east valley wall of the Missouri River[8]. It also thins out rather abruptly in a southerly direction from the Wisconsin-drift border.

Over one hundred samples of the Wisconsin loess have been taken along the five traverses (figure 1). Samples were taken at a depth of from 2 to 3 feet below the top of the C horizon at each of the locations shown on the map; at many of the locations samples were also taken at greater depths. All sampling was done on uneroded ridges or hilltops. A 6 inch soil auger was used for securing samples when suitable road cuts could not be found.

The following tests are being used to evaluate the areal and stratigraphic uniformity of the Wisconsin loess:

1. Liquid limit
2. Plastic limit
3. Plasticity index
4. Shrinkage limit
5. Centrifuge moisture equivalent
6. Field moisture equivalent
7. Hygroscopic moisture (air-dry)
8. Mechanical analysis
9. Specific gravity
10. Field moisture content
11. Field density (in-place)
12. Color, wet and air-dry
13. Textural and engineering classification
14. pH
15. Organic matter content
16. Carbonate content, expressed as percent $CaCO_3$
17. Sulfate content, expressed as percent SO_3
18. Cation exchange capacity (whole soil)
19. Differential thermal analysis
20. Types of exchangeable cations.

These tests have been made on more than fifty of the loess samples at the time of this writing. The data indicate that physical and chemical properties along the north-south first bluff-line traverse (Traverse 1) are remarkably uniform for a natural deposit of soil material. With increasing distance away from the Missouri River valley, however, the properties of samples taken along three of the inland traverses (Traverses 3, 4, 5), reflect a marked increase in plasticity, shrinkage, water-holding capacity, and in-place density; the samples taken along the Wisconsin-drift border (Traverse 2) have not as yet been tested. The data further indicate that this increase is almost wholly due to an increase in the amount of clay in the loess.

On the basis of the mineralogical and chemical data available, the mineralogical nature of the Wisconsin loess seems to be quite uniform; the clay portion being mainly composed of illite- and montmorillonite-type clay minerals with calcium and magnesium as the predominant exchangeable cations. Organic matter and soluble sulfate contents are very low or nonexistent. Aside from variation in amount of clay, the principal variable in the loess appears to be carbonate content. Carbonate percentages, expressed as calcium carbonate, were as high as 18 percent along the first bluff-line traverse and decreased to as low as 1 percent in samples taken near the east boundary of the loess area.

Correlations

The test data for the Wisconsin loess affords an opportunity for correlations of amount of clay and engineering properties, since the amount of clay present in the loess was found to be the major variable responsible for the differences in engineering properties.

In this study clay is defined as the soil particles smaller than 0.002 mm. in equivalent spherical diameter[2]. This definition has been given further scientific justification as a result of mineralogical studies of soils by several investigators[11, 13, 15]. The 0.002-mm. upper limit for clay has been adopted by the International Society of Soil Science and the U. S. Department of Agriculture; it is also used in the MIT system of particle-size classification. Trial correlations in the present study showed that the amount of 0.002-mm. clay present in the loess samples correlated better with engineering properties than the amount of 0.005-mm. clay, which is commonly used as the clay portion of soils in current engineering practice.

The properties to be correlated were determined by means of the following test procedures:

1. Mechanical analysis. ASTM Designation: D422-39 as modified[3].
2. Liquid limit. AASHO designation: T89-49.
3. Plastic limit. AASHO designation: T90-49.
4. Plasticity index. AASHO designation: T91-49.
5. Shrinkage limit. AASHO designation: T92-42.
6. Centrifuge moisture equivalent. AASHO designation: T94-42.
7. Field moisture equivalent. AASHO designation: T93-49.
8. Hygroscopic (air-dry) moisture. ASTM designation: D422-39.
9. Field (in-place) density. The rubber balloon method was used[4].
10. Field moisture content. The method for material that does not contain aggregate larger than ¼ in. was used[4].

The influence of the amount of clay on the several engineering properties was determined by plotting on linear graph paper the value of the engineering property of each loess sample against the sample's percentage of 0.002-mm. clay (figures 2 to 10). All properties, except the field moisture equivalent (figure 7) have an apparent linear relationship with 0.002-mm. clay content. The curves, whose equations are shown on the graphs, were fitted visually by balancing the number of points on either side of the line.

The field-density points (figure 9) represent only the loess densities as measured at a depth of between 2 and 3 feet below the top of the C horizon. This was necessary, because in-place density increases with depth in the loess and clay content does not. The graphs for all other properties represent the loess at the 2 to 3 foot depth and deeper.

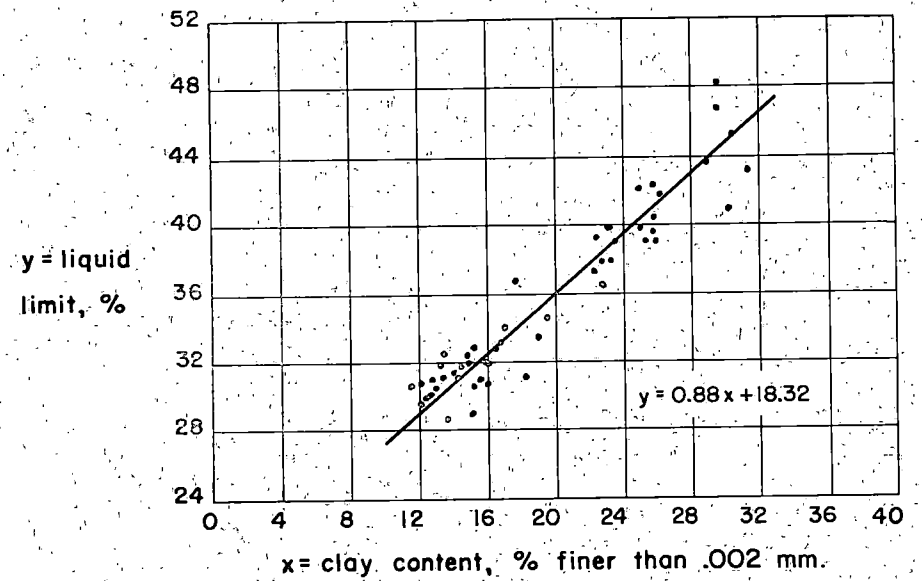

Fig. 2. Influence of clay content on liquid limit.

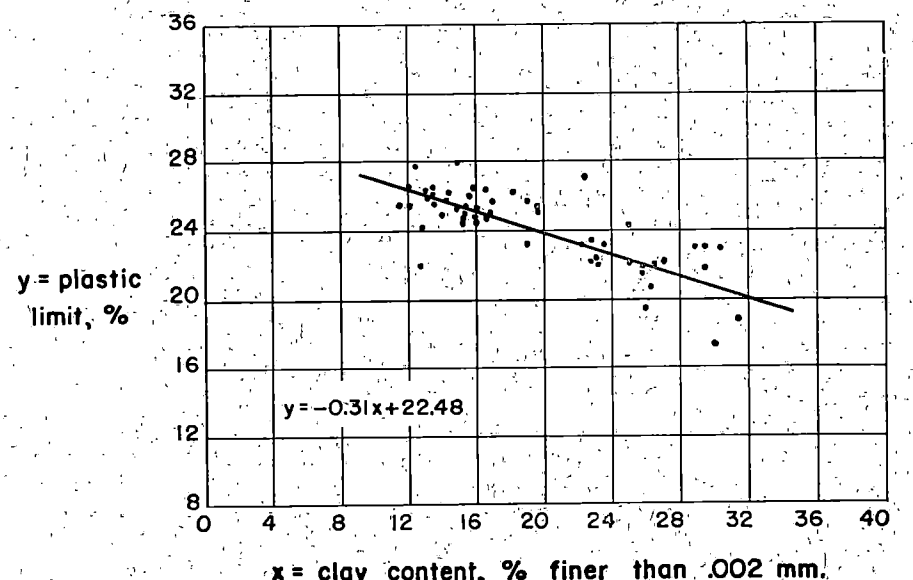

Fig. 3. Influence of clay content on plastic limit.

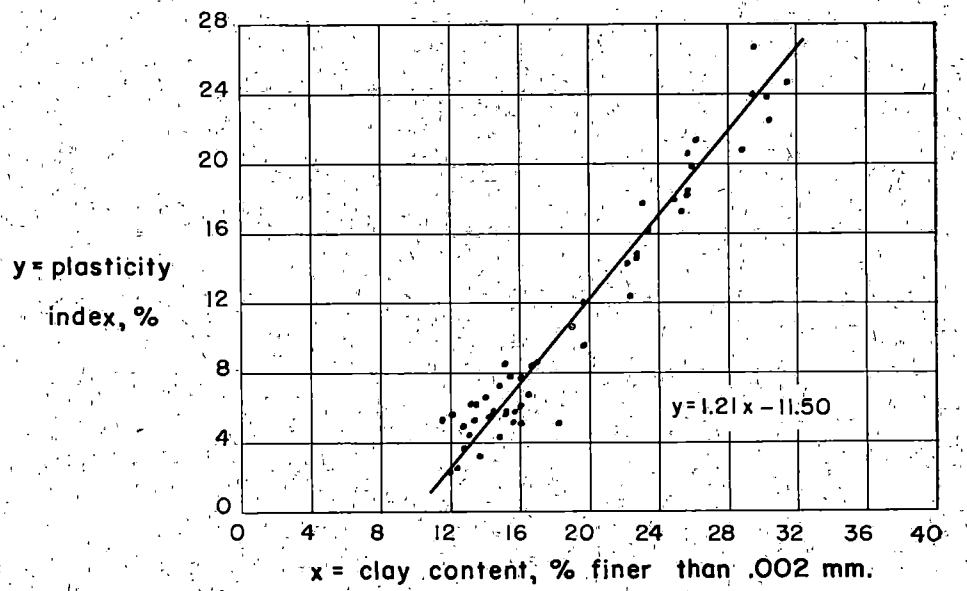

Fig. 4. Influence of clay content on plasticity index.

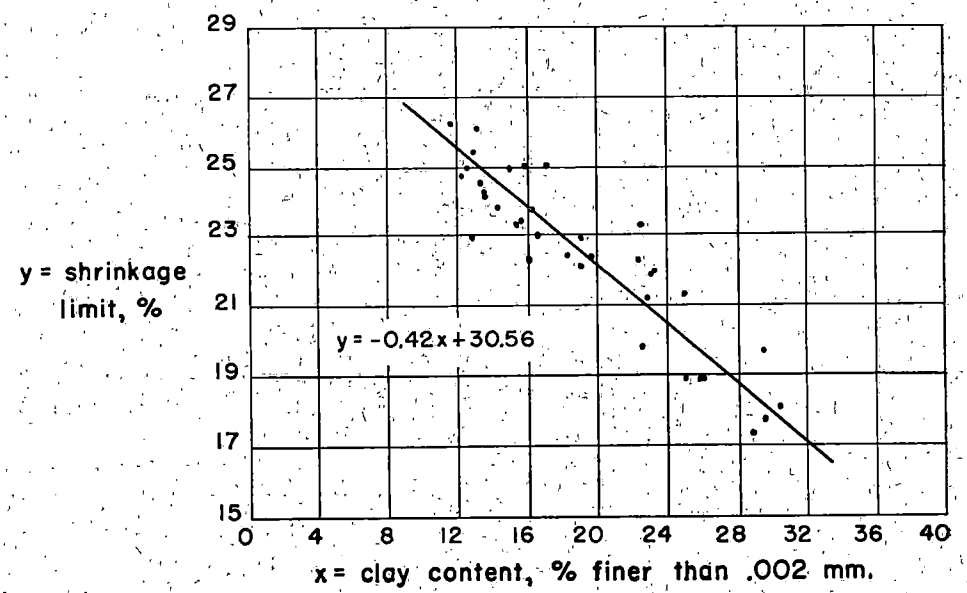

Fig. 5. Influence of clay content on shrinkage limit.

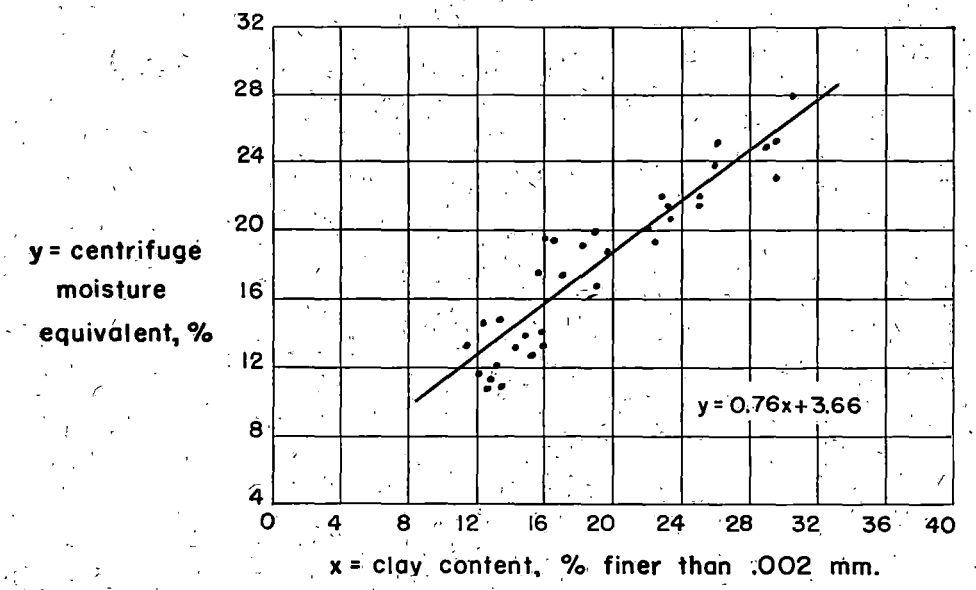

Fig. 6. Influence of clay content on centrifuge moisture equivalent.

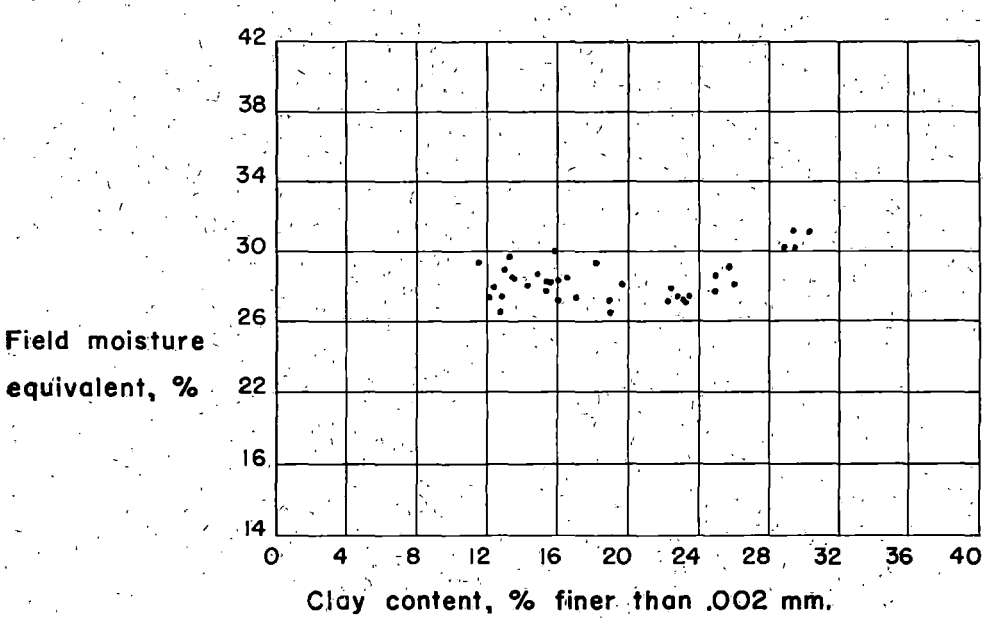

Fig. 7. Influence of clay content on field moisture equivalent.

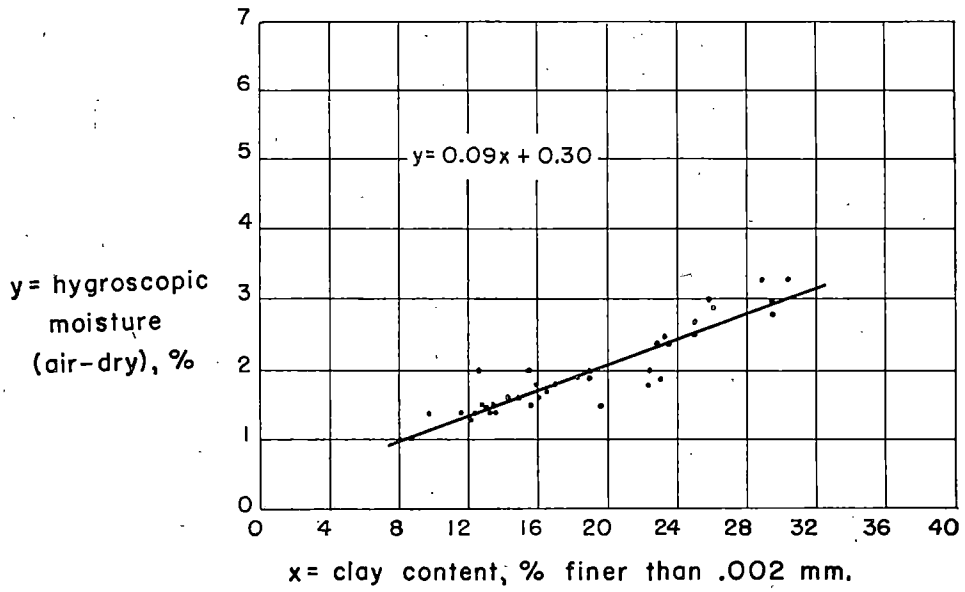

Fig. 8. Influence of clay content on hygroscopic moisture.

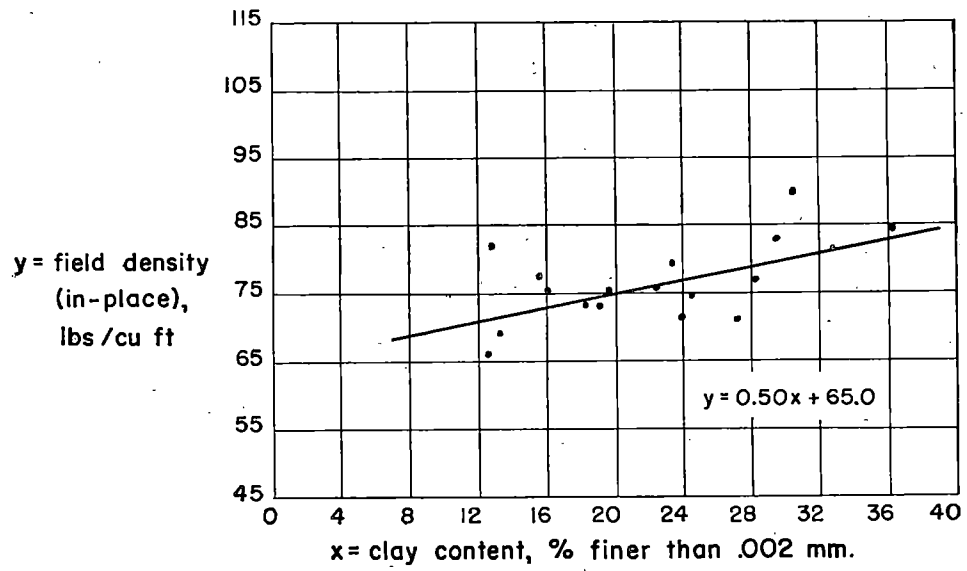

Fig. 9. Influence of clay content on field density.

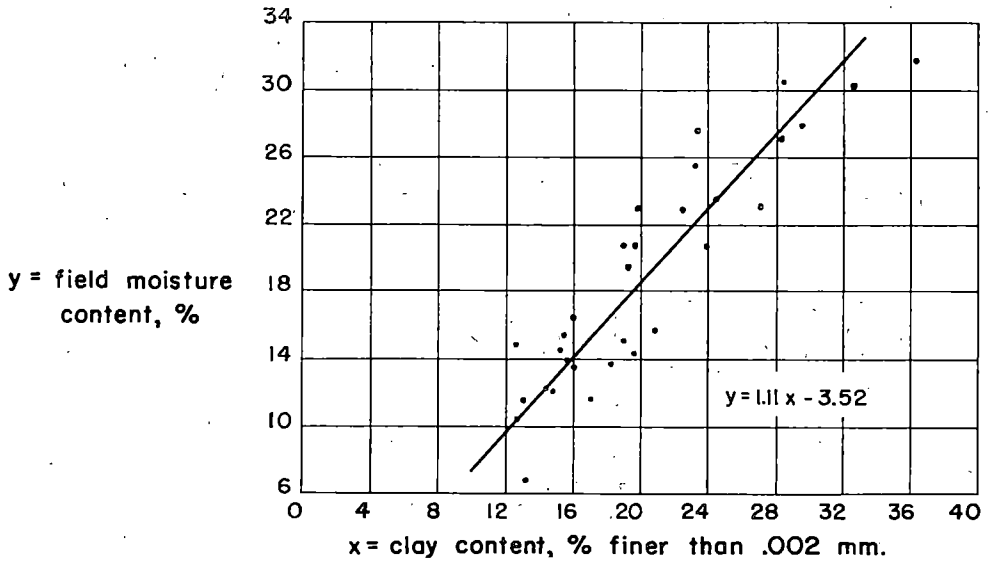

Fig. 10. Influence of clay content on field moisture content.

The results of the correlations show that in a soil system where amount of clay is the major variable, the liquid limit, plasticity index, centrifuge-moisture equivalent, hygroscopic moisture, field-moisture content, and field density[6] are directly proportional to the 0.002-mm. clay content, showing an increase in value with an increase in clay content. The plastic limit and shrinkage limit are also directly proportional to the 0.002-mm. clay content, but show a decrease in value with an increase in clay content. All of these relationships follow the general equation: $y = mx + b$. In this equation y represents the engineering property; x the 0.002-mm. clay content; m the slope of the curve which can be positive or negative, depending upon the engineering property under consideration; b is also a constant, depending upon the same engineering property.

The field moisture equivalent shows no simple relationship to the 0.002-mm. clay content. On the basis of a similar study with British soils, the Road Research Laboratory in England concluded[10]: "The field moisture equivalent varies only slowly as the clay content increases and even for a given type of soil exhibits considerable variability, the cause of which is not known. Even as an index test, its value seems very doubtful." The data of the present study appear to confirm this statement.

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EFFECT OF WETTING TIME ON PLASTICITY INDICES OF CLAY SOILS

by

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(Progress Report, 1960.)

ABSTRACT

The plasticity index has been widely used in soil classification and identification systems. Research and experience have established that the clay portion of a soil and its mineralogical character play an important role in the engineering behavior of the soil. Since water affects the soil system through the clay-water interaction, the effect of the wetting time or "tempering" as reflected in the plasticity index of the soil was investigated.

This study seems to show that Atterberg limits tests for kaolinite clay soils should be run during the 0 to 1 hour wetting time if the maximum plasticity index is sought, but the maximum change in the plasticity index of the same soil is not shown until after a 21 hour wetting period. With montmorillonitic clay soils though, both characteristics, the maximum plasticity index and the maximum change in the plasticity index, are reflected at about the 3 hour wetting period.

CONSISTENCY OF SOILS

The Atterberg limits

The current concept of how the engineering properties of soils are related to the characteristics of those soils when containing varying amounts of moisture began in 1911 when the Atterberg limits were suggested[2]. This theory is that the critical limits of moisture content in soils increase as the moisture content rises from hygroscopicity through the plastic and into the liquid status. The works of other investigators amplified this theory and refined the testing procedures[6].

The ASTM standard consistency tests

The standardization of the definitions of consistency limits and of the testing procedures to quantitatively attain these limits was a natural growth. The American Society for Testing Materials adopted and standardized the procedures[1], and defines the consistency limits as follows:

The liquid limit of a soil is the moisture content, expressed as a percentage of the weight of the oven-dried soil, at the boundary between the liquid and the plastic states. The water content at this boundary is arbitrarily defined as the water content at which two halves of a soil cake will flow together for a distance of ½ inch along the bottom of the groove separating the two halves, when the cup is dropped 25 times for a distance of 1 cm. (0.3937 in.) at the rate of two drops per second.

The plastic limit of a soil is the lowest moisture content, expressed as a percentage of the weight of the oven-dried soil, at the boundary between the plastic and semisolid states. The water content at this boundary is arbitrarily defined as the lowest water content at which the soil can be rolled into threads ⅛ inch in diameter without the threads breaking into pieces.

The plasticity index of a soil is the difference between its liquid limit and its plastic limit.

Standardization, however, does not imply the end of any modification. The recent tendency for rapid methods of test has led to the suggestion of the "one-point method" instead of the "three-point method" of determining the liquid limit of a soil[5]. Accurate duplication of results also requires improved standardization of some details in the design of the apparatus leading, possibly, to the replacement of the liquid limit test by a more rational test based on the measurement of the shear strength of the soil[3].

Soil plasticity and the nature of clay

The plastic or rheological properties of soils do more than provide an excellent basis for classification and identification; they give an idea as to the shear strength of soils. Since the shear strength of soils accrues from the clay content, it becomes evident, in the light of the modern concept of clays, that not only the amount but also the type of clay mineral bears heavily on the value of the plasticity index. Listing the clay minerals in a descending order of surface activity, soils containing montmorillonitic clay have a higher plasticity index than those containing illitic clay, which in turn have a higher plasticity index than those containing kaolinitic clay. Also, if the surface activity of clay particles governs their rate of hydration, it follows that there is a critical period of time after mixing the soil with water during which the plasticity tests should be conducted so that the plastic properties of soils are best reflected in the test data.

INVESTIGATION

Soils

Of the five soils tested, four were from Iowa and one from Maryland; the soils covered a range of characteristics both from the standpoint of texture and plasticity as well as clay content and clay mineral constituents (table I). With the exception of EPK, which was a commercially produced kaolin used as a check on the results obtained with soil number 5, all soils were natural soils.

Preparation of soil samples

The essence of this investigation lay in determining the plasticity indices of representative portions of the five natural soils and EPK after allowing them to stand mixed with water for various periods of time. Since sorption of water by colloids, excepting bentonite, has been shown to be practically complete after 24 hours, it was assumed that any trend due to differential times of wetting would be apparent within 24 hours[6]. Therefore 0, 1, 3, and 21 hour periods of time were chosen for the wetting of the samples before testing for plasticity. By comparison of the plasticity indices of the same soil, obtained from tests performed on portions wetted for different lengths of time, the effects of the wetting time could be observed.

Testing procedures

The tests for the liquid and plastic limits, and the plasticity indices of the soils were conducted according to the procedures outlined in ASTM Designation: D423-54T, Mechanical Method, and ASTM Designation: D424-54T[1]. The amount of water added to the soil for the wetting period was at about that of the liquid limit. The soil was mixed with water for at least

Table I. Description and properties of the soil samples.

Sample No.	1	2	3	4	5	6(EPK)*
Geological description:	Wisconsin-age loess	Kansan-age till	Kansan-age till	Planosol on leached, fine textured Wisconsin-age loess		
Sampling location	Dallas Co., DeSoto, Ia.	Dallas Co., DeSoto, Ia.	Guthrie Co., Guthrie Center, Ia.	Wayne Co., Iowa	Prince George's Co., Md.	
Soil series	Tama	Paleosol	Shelby	Edina	Sassafras	
Horizon	C_1	B_p, C_p		B	C	
Depth, ft.	2-8.2	8.2-18.4	0.9-7.2	2-3	-	
Textural composition,%						
Gravel (2.00 mm)	0	7.0	0.0	0.0	58.0	
Sand (2.00-0.074 mm)	0	26.0	51.0	0.7	23.9	
Silt (0.074-0.005 mm)	60.0	24.0	31.0	36.8	10.1	14.0[f]
Clay (0.005 mm)	40.0	43.0	18.0	62.5	8.0	86.0
Colloids (0.001 mm)	24.0	26.0	11.0	53.5	4.4	35.0
Predominant clay mineral	Montmorillonite[≠]	Montmorillonite[≠]	Montmorillonite[≠]	Montmorillonite[≠]	Kaolinite[&]	Kaolinite
Classification						
Textural**	Silty clay	Clay	Sandy loam	Clay	Gravelly sandy loam	Clay
Engineering (AASHO)	A-7-6(16)	A-7-6(16)	A-4(3)	A-7-6(20)	A-2-6(0)	

*Commercially produced kaolin by the Edgar Plastic Kaolin Co., Edgar, Florida.

[f]All silt-size particles are less than 0.040 mm in diameter.

[≠]By X-ray diffraction analysis[4].

[&]DTA[6].

**From triangular chart developed by U.S. Bureau of Public Roads, but 0.074 mm was used as the lower limit of the sand fraction.

ten minutes. After this the soil mixtures were put into covered containers and stored in a humidifier for designated periods of time. Then the soil specimens were tested.

TABLE II. SUMMARY OF ATTERBERG TEST RESULTS

Sample No. and predominant clay mineral	Time tests performed after mixing, hr.	Liquid limit, %	Plastic limit, %	Plasticity index, %
1. Montmorillonite	0	48	26	22
	1	51	25	26
	3	50	25	25
	21	50	25	25
2. Montmorillonite	0	56	24	32
	1	56	25	31
	3	57	24	33
	21	55	24	31
3. Montmorillonite	0	25	18	7
	1	23	19	4
	3	25	18	7
	21	23	18	4
4. Montmorillonite	0	77	36	41
	1	74	35	39
	3	76	35	41
	21	76	36	40
5. Kaolinite	0	35	24	11
	1	35	23	12
	3	35	23	10
	21	34	26	8
6. (EPK) Kaolinite	0	55	34	21
	1	59	37	22
	3	55	36	19
	21	52	36	16

Four determinations were used for the construction of the flow curve for the liquid limit. The average of three values was taken as the plastic limit of the soil. Following the ASTM procedures, all water contents have been reported to the nearest whole number in the data (table II).

TABLE III. EFFECT OF WETTING TIME ON PLASTICITY

Soil sample No.	Changes in L.L. between wetting times of 0 and			Changes in P.L. between wetting times of 0 and			Changes in P.I. between wetting times of 0 and		
	1 hr.	3 hr.	21 hr.	1 hr.	3 hr.	21 hr.	1 hr.	3 hr.	21 hr.
1	+3	+2	+2	−1	−1	−1	+4	+3	+3
2	0	+1	−1	+1	0	0	−1	+1	−1
3	−2	0	−2	+1	0	0	−3	0	−1
4	−3	−1	−1	−1	−1	0	−2	0	−1
5	0	0	−1	−1	+1	+2	+1	−1	−3
6(EPK)	+4	0	−3	+3	+2	+2	+1	−2	−5
Increases	2	2	1	3	2	2	3	2	1
Decreases	2	1	5	3	2	1	3	2	5
No change	2	3	0	0	2	3	0	2	0

DISCUSSION OF RESULTS

Time of wetting or "tempering," appears from the data to alter to some degree the plastic properties of soils (figure 1, table III). Since the plasticity index of a soil is of importance in engineering, the influence of time of

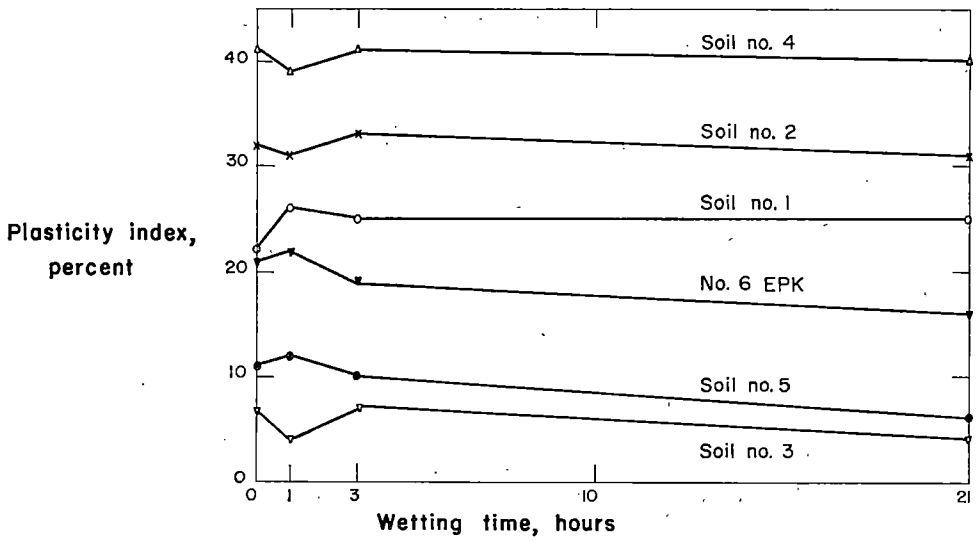

Fig. 1. Effect of wetting time on the plasticity indices.

wetting may best be interpreted in terms of the clay constituents of a soil and on the basis of changes in the plasticity index.

The three soils, 2, 3, and 4, containing montmorillonitic clay in amounts varying from 18 to 62.9 percent displayed the same trend: the plasticity index decreased at the end of 1 hour wetting, increased to a maximum at the 3 hour wetting, and tended to decrease as the 21 hour wetting period was reached. The four soil, No. 1, also a montmorillonitic clay, behaved in a different way, although it contained exactly the average amount of clay (40 percent) when 18 and 62.5 percent are taken as the extreme values. It manifested the maximum plasticity index value at the 1 hour wetting period, decreased slightly towards the 3 hour wetting period and seemed to have retained this value as the 21 hour wetting period was reached.

In spite of the difference of behaviour between the first three soils, 2, 3, and 4, on one hand and soil No. 1 on the other, it is evident that all four soils required a wetting period of more than 1 hour before displaying their maximum plasticity index values. In fact, discounting the small difference between the 1 hour and 3 hour plasticity index values in soil No. 1, it will be safe to state that the maximum plasticity index of the montmorillonitic clay soils used in this experiment occurred between the 1 and 3 hour wetting

period, and possibly near the 3 hour wetting period. It is also of equal importance to note that the maximum difference in the plasticity index of the montmorillonitic soils occurred during the 0 to the 3 hour wetting period.

The kaolinitic soils, No. 5 and No. 6 (EPK), reacted similarly within the group but differently than the montmorillonitic soils in that their plasticity indices reached maximum at the 1 hour period, and minimum at the 21 hour period.

From the results of this investigation it appears that, if the determination of the maximum plasticity index of a clay soil is required, the 1 hour wetting period should be used for the kaolinitic soils, and the 3 hour wetting period for the montmorillonitic soils. The longer time required by the montmorillonitic soils is attributed to their higher specific surface and higher surface activity compared with the kaolinitic soils.

The prolonged wetting period of 21 hours does not seem to change drastically the plastic properties of montmorillonitic clays over those determined at the 3 hour period. The 21 hour plasticity index values seem to be not very remote from the average plasticity index, when the average is calculated from the four wetting periods. With the kaolinitic soils, however, the 21 hour wetting period is more critical than it is with montmorillonitic soils, because it seems to represent not only the minimum plasticity index value but also it is far from being close to the average value.

If reduction of the plasticity index is taken as an indication of increasing aggregation, and conversely, an increase in plasticity index is taken as increasing dispersion, then the results are indicative of the long term role as a flocculating agent of water when it is added in controlled amounts. The influence of water, when short and long wetting periods are compared, seems to be more pronounced with kaolinitic soils than with montmorillonitic soils. It is possible that the kaolinitic clay particles attain some degree of orientation upon the addition of water and subsequent manipulation, but with time they lose this orientation. This seems to hold true for montmorillonitic clay particles except that the loss of orientation with time is small.

Although the applicability of the conclusions drawn from this investigation does not seem to be questionable, further research may help in substantiating the facts brought out in this work.

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THE RELATION BETWEEN SOIL MOISTURE TENSION AND THE CONSISTENCY LIMITS OF SOILS

by

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(Progress Report, 1955.)

ABSTRACT

Soil physicists use the capillary potential as a means of expressing the energy of attraction of soil for water. The so-called tension-moisture curves are used to characterize soils and to display soil moisture relations. By use of equipment now commercially available tension values and moisture content data can be determined for a large number of samples at one time.

Procedures are given herein for preparing the soil and for determining the soil moisture tension curves. Data are presented showing the relationship between tension values and the liquid and plastic limits of soils in the textural groups of silty loam, silty clay loam, silty clay, and clay. Tension values for at least 32 duplicate samples can be obtained in a 24 hour period for the liquid limit.

By the use of the correlation data presented, much time can be saved in estimating the consistency limits.

INTRODUCTION

The consistency limits of soils have been used widely in soil engineering for the classification and identification of soils. The American Association of State Highway Officials soil classification, the unified soil classification of the U. S. Corps of Engineers, and various other systems utilize Atterberg's limits. These limits are often used directly in specifications for mechanical stabilization, for determining the kind of soil to be used in fills, and in the design of flexible pavements. Thousands of these tests are made each year by soil engineering organizations. A disadvantage of procedures currently available for determining consistency limits is the time consumed in completing a test. For instance, it takes the average laboratory technician at least an hour to arrive at a value for the liquid limit, and nearly as long to determine the plastic limit.

TABLE I. SOILS USED IN THE INVESTIGATION

Soil No.	L.L.	P.L.	P.I.	Sand	Silt	Clay <5μ	Textural Classification
108-1	32.1	21.6	10.5	2.3	71.1	26.6	Silty clay loam
108-2	47.4	19.8	32.4	0.5	57.5	42.0	Silty clay
119-1	38.5	24.1	14.4	0.9	63.9	35.2	Silty clay
117-2	47.7	19.0	29.8	1.1	57.3	41.6	Silty clay
114-5	29.1	19.2	9.9	6.3	75.1	18.6	Silty loam
117-7	31.0	20.3	13.3	0.6	74.9	24.5	Silty clay loam
114-7	26.6	19.2	7.4	8.1	74.7	17.2	Silty loam
108-4	35.6	19.9	17.4	1.0	71.2	27.8	Silty clay loam
119-5	38.4	17.2	21.2	0.9	67.1	32.0	Silty clay
110-5	31.0	21.7	9.3				Silty clay loam
202-1	30.4	22.7	5.9	1.4	74.3	24.3	Silty clay loam
207-1	39.3	29.0	10.3	1.2	70.4	28.4	Silty clay loam
212-2	43.9	21.9	22.0	4.2	58.3	37.5	Silty clay
227-2	45.5	22.3	25.7	0.8	62.6	36.6	Silty clay
202-6	27.7	22.7	5.0	0.6	80.2	19.2	Silty loam
211-5	39.9	23.0	16.9	0.4	65.7	33.9	Silty clay
217-4	41.8	22.3	19.5	0.4	64.0	35.6	Silty clay
227-5	24.1	21.0	3.1	0.5	85.0	14.5	Silty loam
211-7	26.8	23.0	3.8	1.6	79.6	18.8	Silty loam
LF-1	57.4	19.9	37.5	7.7	48.2	44.1	Clay
LF-2	73.7	51.2	24.0	3.4	12.0	84.6	Clay
LF-3	31.8	24.6	7.2	0.7	78.3	21.0	Silty clay loam
LF-4	33.0	22.4	10.6	7.7	55.7	36.6	Silty clay
M38-1	32.7	27.1	5.6	3.78	79.82	16.4	Silty loam
M25-1	36.5	31.3	5.2	4.61	78.29	17.1	Silty loam
M83-1	40.9	34.7	6.2	4.98	78.92	16.1	Silty loam
43	35	20	16	25	39	33	Clay
61	56	24	32	6	42	52	Clay
62	46	20	26	7	49	43	Clay
65	70	27	43	10	32	57	Clay
84	42	21	21	18	40	41	Clay
86	43	21	22	24	37	38	Clay
89	40	22	18	24	37	39	Clay
94	55	25	30	4	43	53	Clay
43½	51.9	20.8	33.4	0.7	60.2	39.4	Clay
Luton	71.0	24.5	47	2.11	27.89	7.0	Clay
2963	70	36	34	19	26	55	Clay
Gumbotil	66.8	21.9	48.7	8.0	31	61	Clay
V-1	41.4	20.7	20.7	0.9	66.6	32.5	Silty clay
V-4	51.1	20.1	31.0	0.4	56.6	43.0	Clay
IV-1	47.7	22.4	25.3	1.3	60.6	38.1	Silty clay
IV-5	40.6	18.8	21.8	0.7	66.0	33.3	Silty clay
I-16	30.3	21.2	11.4	3.1	72.9	24.0	Silty clay loam
IV-14	36.1	22.4	16.5	0.5	74.7	24.8	Silty clay loam
II-7	30.2	24.4	5.8	2.1	84.3	13.6	Silty loam
IV-15	35.3	21.4	13.9	0.7	76.3	23.0	Silty clay loam
I-21	26.8	21.2	5.6	4.7	81.2	14.1	Silty loam
15-2	30.6	24.0	4.5	1.0	83	16	Silty loam
55-1	29.6	27.3	2.3	1.3	85.3	13.4	Silty loam
47-1	34.7	25.1	9.6	1.5	76.3	22.4	Silty clay loam
48-2	31.6	19.7	11.9	0.7	71.3	28.0	Silty clay loam
34-1	46.8	22.8	24.0	0.9	64.1	35.0	Silty clay
37-2	39.2	19.5	19.7	0.2	67.3	32.5	Silty clay
43-1	39.8	16.7	23.1	10.9	56.8	32.3	Silty clay
46-1	54.7	22.0	32.7	0.5	56.0	43.5	Clay
71-2	38.4	22.7	15.7	0.8	72.2	27.0	Silty clay loam
59-1	48.5	20.0	28.5	0.5	59.1	40.4	Silty clay
15-1	31.9	24.0	6.2	3.4	79.4	17.0	Silty loam

Herein is a method for estimating the consistency limits by relating them to soil moisture tension values determined by porous plate equipment and pressure membrane apparatus[2].

SOILS USED

Fifty-six different soils, most of which were Iowa soils, all ranging in texture from silty loam to clay, were studied (table I).

APPARATUS USED FOR DETERMINING SOIL MOISTURE TENSION

Soil moisture tensions up to twenty pounds per square inch were obtained on the porous plate apparatus[2, 3]. The apparatus has three ways for measuring the applied air pressure. In the investigation a water manometer was used for pressures up to twenty inches of water, a pressure gage graduated from zero to two hundred inches of water was used for the range of twenty to two hundred inches, and a pressure gage with a maximum reading of thirty pounds per square inch was used beyond two hundred inches of water and up to twenty pounds per square inch. The air pressure was supplied from an ordinary laboratory compressed air line and controlled by two valves. Valve 1 supplied air to valve 2 at a pressure of approximately forty pounds per square inch. Valve 2 is a sensitive pressure reduction valve which maintains a constant pressure in the porous plate apparatus. The apparatus has four porous ceramic plates. Soil moisture tensions greater than twenty pounds per square inch were determined by using a Richards pressure membrane apparatus which can determine soil moisture tensions up to fifteen atmospheres.

Brass rings, one and three quarters inches in diameter and one-half inch high, were placed on the porous plate to contain the soil sample. A maximum of eighteen rings of this size can be placed on each of the four plates, a total of seventy-two samples in the apparatus at one time. If duplicate samples are used for each soil type, thirty-six soils can be tested at one time.

Rubber rings, approximately the same size as the brass rings, were used on the pressure membrane apparatus. In this apparatus thirty rings or fifteen duplicate soil samples can be investigated at one time.

PREPARATION OF SOIL FOR DETERMINING SOIL MOISTURE TENSION

Each soil sample for which a soil moisture tension value was desired was saturated essentially in accordance with procedures outlined in USDA Handbook No. 60[4]. Specifically the procedure used was as follows:

> Approximately thirty grams of a representative sample of the soil was placed in one-half pint fruit jar, and sufficient water was added at one time to bring it nearly to the saturation point. Where a large number of samples were being prepared, as many jars as were necessary were lined up in a row, and the soil was placed in them. Sufficient water was then added to each sample to bring it nearly to the saturation

point. Each sample in turn was then brought to the saturation point by slowly adding more water and mixing with a spatula.

To determine the end point of saturation, the soil mass was transferred to one side of the jar. If the soil slowly flowed when the jar was tipped to an angle approximately 60 degrees with the horizontal, saturation was assumed. The jars were then capped, and the samples were allowed to stand for an hour or more, after which they were again checked for saturation. The saturated soil was then placed in the rings on the saturated porous plates.

The saturation process must be followed carefully, since the soil moisture tension at each of the limits is affected by the initial moisture content.

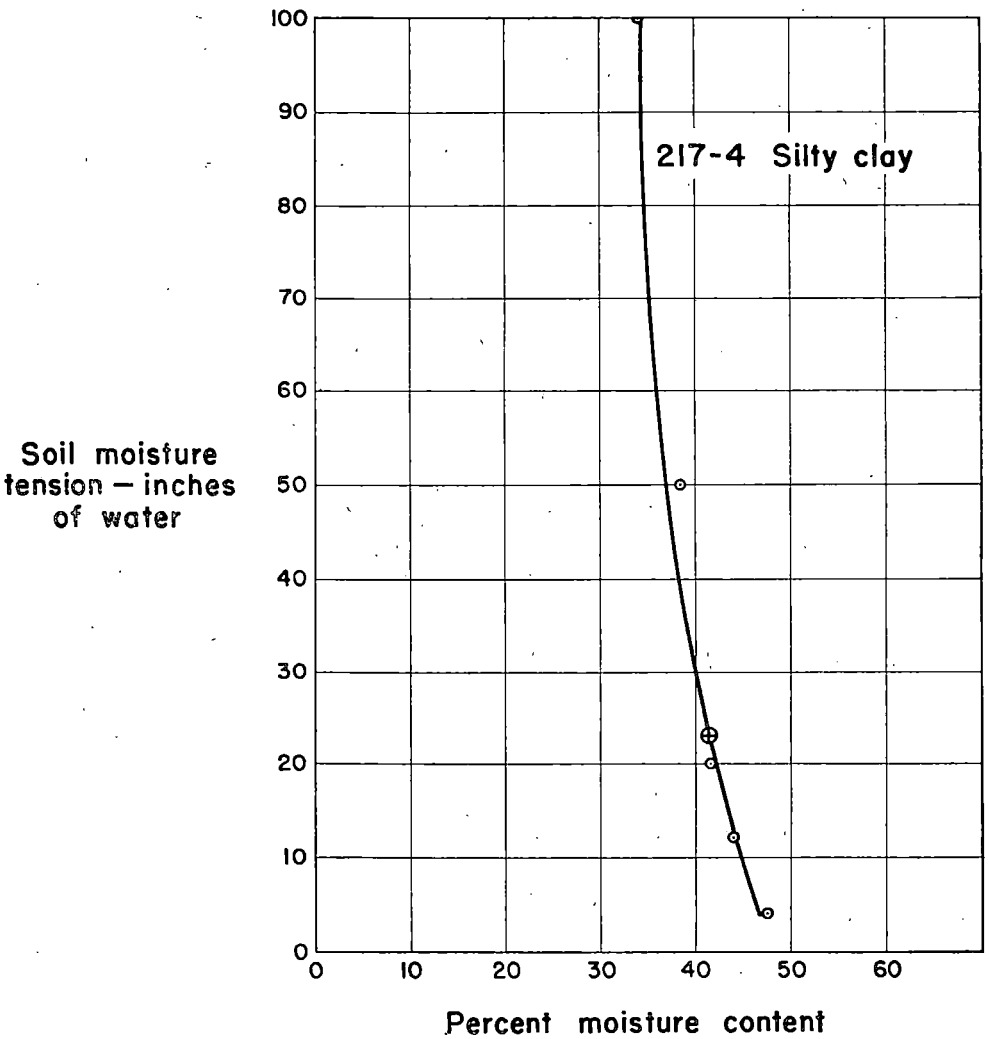

Fig. 1. Relationship between soil moisture tension and moisture content.

RELATIONSHIP BETWEEN SOIL MOISTURE TENSION AND THE CONSISTENCY LIMITS

Preliminary tests indicated that more useful results could be obtained from this study if the relationship between soil moisture tension and the consistency limits was established for each textural group. The study was then conducted with this objective in mind. The textural groups available for investigation, according to the U. S. Bureau of Public Roads textural classification system, were silty loam, silty clay loam, silty clay, and clay. Preliminary tests indicated that the soil moisture tension corresponding to the liquid limit varied from about five to forty inches of water pressure for all soils, and the moisture tension corresponding to the plastic limit varied from about 160 to 1700 inches of water.

With this information, moisture contents corresponding to four, eight, twelve, twenty, fifty, and one hundred inches of water were determined for all soils for the liquid limit study. Two trials were made by different operators for each pressure with duplicate samples for each trial. The averages of these two trials were then used to plot a curve with soil moisture tension or applied pressure as a function of the moisture content (figure 1). From these curves for each soil, the most probable soil moisture tension within a textural classification that would yield a moisture content equivalent to the liquid limit was selected.

The procedure used to estimate the appropriate soil moisture tension was to select various tensions in the vicinity of the desired one by direct observation. Moisture contents were determined for each tension for all soils within the textural group. The deviations of the liquid limit from the moisture content corresponding to a certain tension for all soils were determined. Aver-

TABLE II. TABULAR ANALYSIS OF SILTY CLAY FOR THE LIQUID LIMIT

Soil Designation	L.L.	% H_2O for 20 in. Ten.	Dev. from L.L.	% H_2O for 15 in. Ten.	Dev. from L.L.	% H_2O for 10 in. ten.	Dev. from L.L.
217-4	41.8	42.2	+0.4	43.4	+1.6	42.2	+0.4
211-5	39.9	40.4	+0.5	41.7	+1.8	43.0	+3.1
227-2	48.0	44.8	−3.2	46.0	−2.0	47.3	−0.7
34-1	46.8	45.3	−1.5	46.5	−0.3	48.0	+1.2
V-1	41.4	38.2	−3.2	39.3	−2.1	40.5	−0.9
37-2	39.2	40.3	+1.1	41.4	+2.2	42.5	+3.3
212-2	43.9	41.2	−2.7	42.3	−1.6	43.5	−0.4
119-5	38.4	38.4	0.0	39.4	+1.0	40.5	+2.1
119-1	38.5	39.9	+1.4	40.8	+2.3	41.6	+3.1
117-2	47.7	45.6	−2.1	46.8	−0.9	48.2	+0.5
IV-5	40.6	41.4	+0.8	40.3	−0.3	44.0	+3.4
LF-4	33.0	35.2	+2.2	36.0	+3.0	37.0	+4.0
43-1	39.8	35.3	−4.5	36.7	−3.1	38.4	−1.4
IV-1	47.7	45.7	2.0	46.9	−0.8	48.1	+0.4
Mean			1.83		1.64		1.78
Algebraic Mean			−0.63		+0.057		+1.28

age deviations and average algebraic deviations were determined for each tension for all soils. In a given textural group, the soil moisture tension which yielded the minimum algebraic and average deviation was selected (table II).

In determining the soil moisture tension corresponding with the plastic limit, soil moisture tension curves were obtained by finding moisture contents corresponding with pressures of 100, 200, 405, 1383 and 2767 inches of water pressure. Soil moisture tensions corresponding to the liquid limit were estimated from these curves for each textural classification in exactly the same manner as described above. This analysis indicated that the tensions for the various textural groups should be approximately as indicated:

TEXTURAL GROUP	TENSION FOR LIQUID LIMIT	TENSION FOR PLASTIC LIMIT
Silty loam	60 inches of water	168 inches of water
Silty clay loam	60 inches of water	415 inches of water
Silty clay	15 inches of water	913 inches of water
Clay	6 inches of water	1650 inches of water

To check the above results, two samples of each soil were prepared in accordance with the above procedure, then they were subjected to the appropriate pressure (tables III - IX). The average and the algebraic mean

TABLE III. MOISTURE CONTENT FOR 60 INCHES MOISTURE TENSION COMPARED WITH THE LIQUID LIMIT FOR SILTY LOAM AND SILTY CLAY LOAM SOILS

Soil Type	Trial No. 1	Trial No. 2	Average	L.L.	Deviation
114-7	24.8	25.8	25.2	26.6	+1.4
211-7	27.3	26.6	26.9	26.8	−0.1
55-1	30.2	29.7	29.9	29.6	−0.3
I-21	28.0	28.0	28.0	26.8	−1.2
15-2	34.2	34.4	34.3	30.6	−3.7
15-1	33.10	33.1	33.1	31.9	−1.2
202-6	27.6	27.4	27.5	27.7	+0.2
II-7	33.2	31.9	32.5	30.2	−2.3
114-5	28.2	27.0	27.6	29.1	+1.5
227-5	25.8	26.4	26.1	24.1	−2.0
207-1	40.5	42.1	41.3	39.3	−2.0
108-4	33.2	34	33.6	35.6	+2.0
LF-3	32.9	32.5	32.7	31.8	−0.9
110-5	29.5	30.0	29.7	31.0	+1.2
108-1	31.2	31.1	31.1	32.1	+0.9
202-1	33.1	32.9	33.0	30.4	−2.6
IV-15	34.0	33.3	33.7	35.3	+1.6
47-1	34.4	35.4	34.9	34.7	−0.2
IV-14	36.7	35.6	36.1	36.1	0.0
I-16	32.7	31.9	32.3	30.3	−2.0
48-2	34.8	34.9	34.9	31.6	−3.3
117-7	28.8	28.6	28.7	31.0	+2.3

Average Deviation: 1.50
Average Algebraic Deviation: −0.49

TABLE IV. MOISTURE CONTENT FOR 15 INCHES MOISTURE TENSION COMPARED WITH THE LIQUID LIMIT FOR SILTY CLAY SOILS

Soil Type	Trial No. 1	2	3	4	Average	L.L.	Deviation
108-2	43.3	42.7	45.1	45.1	44.1	47.4	+3.3
IV-1	44.8	44.6	48.1		45.8	47.7	+1.9
119-5	40.3	38.7	42.3	43.2	41.1	38.4	−2.7
V-1	36.3	37.3	39.4		37.7	41.4	+3.7
LF-4	36.0	36.2	38.0	37.8	37.0	33.0	−4.0
IV-5	40.2	42.1	42.7	43.2	42.1	40.6	−1.5
37-2	42.0	42.7	43.3	44.0	43.0	39.2	−3.8
59-1	44.0	43.6	46.4	46.3	45.1	48.5	+3.4
211-5	39.0	40.5	42.9	43.5	41.5	40.0	−1.5
34-1	46.8	45		45.2	45.6	46.8	+1.2
119-1	42.0	41.6		45.6	43.0	38.5	−4.5
43-1	38.4	37.6	37.6	41.3	38.7	39.8	+1.1
227-2	43.5	42.1	45.9	44.6	44.0	45.5	+1.5
212-2	43.3	42.2	42.5	42.8	42.7	43.9	+1.2
117-2	44.3	44.5	45.7	46.8	45.3	47.7	+2.4
217-4	44.4	45.8			45.1	41.8	−3.3
				Average Deviation:			2.56
				Average Algebraic Deviation:			−0.10

deviations from the liquid limit determined by conventional method are indicated for each.

This recheck indicated that the selected tensions are fairly satisfactory for the soils tested, and that the deviations of the moisture contents corresponding to the tension values agree quite well in the majority of cases with the consistency limits determined by standard procedures.

Discussion

The results show that moisture tensions of soils saturated according to the procedure presented are not unique at moisture contents corresponding to the liquid or the plastic limit but vary with the textural classification of the soils. One might be led to believe at first that this places a severe restriction on the method; however, it may be seen that in the liquid limit the range in tensions corresponding to the liquid limit is not really so large. For textural classifications varying from clay to silty loam, the soil moisture tension corresponding to the liquid limit varies from six to sixty inches of water pressure. For the clays to silty clays, the variation is from six to fifteen inches of water pressure. Within the latter tension range, the change in moisture content with change in tension does not appear to be sufficiently great to cause excessive deviations of the liquid limit from the moisture content corresponding to the group tension. The lighter textured soils, such as the silty clay loams and silt loams, can range widely in the soil moisture tension without an appreciable change in the moisture content.

The method should be less reliable for soils which are intermediate between one textural group and another than for those falling well within the

group. However, when one considers that the consistency limits as determined by conventional methods are not precise and may vary by several percent, depending on the operator and other uncontrolled variables, the deviations observed are tolerable. In the results for the plastic limit, deviations of the moisture contents corresponding to the soil moisture tension for a textural group are quite large for the silty loams and silty clay loams. The increased deviation might be expected; since determination of the plastic limit in silty soils by the conventional methods is subject to a wider varia-

TABLE V. MOISTURE CONTENT FOR 6 INCHES MOISTURE TENSION COMPARED WITH THE LIQUID LIMIT FOR CLAY SOILS

Soil Designation	Trial No. 1	Trial No. 2	Average	L.L.	Deviation
Gumbotil	65.0	65.0	65.0	66.8	+1.8
LF-1	54.2	58.4	56.3	51.4	−1.1
61	56.1	55.7	55.9	56	+0.1
46-1	51.5	50.9	51.2	54.7	+3.5
Edina	70.6	72.6	71.1	71	−0.1
84	44.5	43.3	43.9	42	−1.9
43	34.6	34.9	34.8	35	+0.2
43½	49.7	49.9	49.8	51.5	+1.7
62	48.1	47.1	47.6	46	−1.6
Luton	69.4	69.3	69.3	71	+1.7
89	42.9	43.9	43.4	40	−3.4
94	60.6	60.7	60.6	55	−5.6
65	69.6	69.1	69.3	70	+0.7
86	42.7	44.3	43.5	43	−0.5
LF-2	72.0	71.0	71.5	73.7	+1.2
V-4	48.0	48.0	48	51	+3.0

Average Deviation: 1.75
Average Algebraic Deviation: 0.00

TABLE VI. MOISTURE CONTENT FOR 168 INCHES MOISTURE TENSION COMPARED WITH THE PLASTIC LIMIT FOR SILTY LOAM SOILS

Soil Designation	Trial No. 1	Trial No. 2	Trial No. 3	Trial No. 4	Mean	P.L.	Deviation
202-6	24.2	25.1	24.8	24.4	24.6	22.7	−1.9
211-7	24.2	23.8	24.4	24.1	24.1	23.0	−1.1
114-5	22.8	23.1	23.4	23.5	23.2	19.2	−4.0
15-2	18.6	20.4	18.9	19.8	19.3	24	+4.7
II-7	19.3	19.2	21.1	21.0	20.2	24.4	+4.2
I-21	16.7	16.1	17.1	17.9	16.9	21.1	+4.2
15-1	21.6	20.8	20.5	21.1	21.0	24.0	+3.0
55-1	25.8	26.0	24.5	24.5	25.2	27.3	+2.1
114-7	23.0	23.2	22.6	22.7	22.9	19.2	−3.7
M25-1	35.8	37.2	.....	.....	36.5	31.3	−5.2
M38-1	31.8	33.7	.....	.....	32.7	27.1	−5.6
M83-1	32.3	33.6	.....	.....	32.9	34.7	+1.8

Average Deviation: 3.46
Average Algebraic Deviation: −0.13

tion than in more plastic soils. The results of the check test for both silty clays and clays at the plastic limit appear to be quite satisfactory.

TABLE VII. MOISTURE CONTENT FOR 415 INCHES WATER PRESSURE MOISTURE TENSION COMPARED WITH THE PLASTIC LIMIT FOR SILTY CLAY LOAM SOILS

Soil Designation	Trial No. 1	2	3	4	Average	P.L.	Deviation
117-7	22.7	23.1	23.3	23.3	23.1	20.3	−2.8
48-2	20.6	20.0	21.2	20.9	20.8	19.7	−1.1
I-16	23.4	23.4	23.8	23.9	23.6	21.2	−2.4
IV-14	24.4	24.6	26.8	26.6	25.6	22.4	−3.2
47-1	18.9	18.8	21.8	21.8	20.6	25.1	+4.5
IV-15	18.6	19.9	22.0	22.2	20.8	21.4	+0.6
202-1	18.5	18.9	19.9	19.9	19.3	22.7	+3.4
108-1	20.3	19.2	22.2	22.4	21.0	21.6	+0.6
110-5	21.8	22.1	21.6	22.0	21.9	21.7	−0.2
LF-3	15.3	15.7	18.9	18.6	17.1	24.6	+7.5
108-4	24.6	24.7	24.3	24.7	24.6	19.9	−4.7
207-1	21.0	21.9	23.3	23.1	22.3	29.0	+6.7
					Average Deviation:		3.14
					Average Algebraic Deviation:		+0.74

TABLE VIII. MOISTURE CONTENT FOR 913 INCHES WATER PRESSURE MOISTURE TENSION COMPARED WITH THE PLASTIC LIMIT FOR SILTY CLAY SOILS

Soil Designation	Trial No. 1	2	Average	P.L.	Deviation
212-2	20.0	18.2	19.1	21.9	+2.8
227-2	19.1	18.2	18.7	21.0	+2.3
43-1	15.2	15.0	15.1	16.7	+1.6
119-1	17.5	16.4	17.0	24.1	+7.0
117-2	22.6	23.6	23.1	19.0	−4.1
34-1	19.8	20.6	20.2	22.8	+2.6
59-1	24.3	23.2	23.7	20.0	−3.7
LF-4	17.9	17.9	17.9	22.4	+4.5
V-1	17.5	17.2	17.4	20.7	+3.3
108-2	21.7	21.6	21.6	19.8	−1.8
IV-5	18.6	18.6	18.6	18.8	+0.2
IV-1	22.1	22.0	22.1	22.4	+0.3
119-5	17.5	17.1	17.3	17.2	−0.1
37-2	17.5	18.6	18.1	19.5	+1.4
217-4	19.1	19.6	19.3	22.3	+3.0
			Average Deviation:		2.58
			Average Algebraic Deviation:		+1.29

The consistency limits, determined by conventional procedures and used for comparison in this study, were determined by several different operators. It should therefore be expected that there would be some variation of these limits from the actual ones. Table X shows the results of the Mississippi Valley Group Cooperative Test on the consistency limits for one soil. Carefully prepared samples of the soil were sent to the highway soil laboratories of ten different states and the soil laboratory of the U. S. Bureau of

Public Roads. Each in turn determined the limits and reported the average of three trials. As noted in the table, the liquid limit varied from 26.2 to 20.85, with an average of 24.3. The plastic limit varied from 16.0 to 11.56 with an average of 13.8.

TABLE IX. MOISTURE CONTENT FOR 1650 INCHES WATER PRESSURE MOISTURE TENSION COMPARED WITH THE PLASTIC LIMIT FOR LEAN AND MEDIUM CLAY SOILS

Soil Designation	Trial No. 1	Trial No. 2	Average	P.L.	Deviation
84	21.4	21.3	21.3	21	−0.3
61	24.9	24.9	24.9	24	+0.9
89	21.4	21.0	21.2	22	+0.8
65	28.8	29.0	28.9	27	−1.9
62	21.3	21.4	21.4	20	−1.4
94	27.4	27.4	27.4	25	−2.4
43	17.5	17.7	17.6	20	+2.4
46-1	24.0	24.7	24.3	22	−2.3
LF-1	22.2	22.9	22.5	19.9	−2.6
V-4	22.4	22.3	22.4	20.1	−2.3
Gumbotil	29.0	28.6	28.8	21.9	−6.8
43½	24.8	24.8	24.8	20.8	−4.0

Average Deviation: 2.34
Average Algebraic Deviation: −1.66

TABLE X. MISSISSIPPI VALLEY GROUP COOPERATIVE TEST ON BINDER SOIL

Organization	Liquid Limit	Plastic Limit	P.I.
USBPR	25	13	12
Nebraska	26.2	14.3	11.9
Minnesota	24.9	13.1	11.8
Missouri	24.6	13.0	11.6
Wisconsin	26.2	16.0	10.2
Illinois	20.85	11.56	9.29
South Dakota	23.51	14.8	8.71
Arkansas	25.0	13.6	11.4
Michigan	23.3	13.9	9.4
Oklahoma	22.9	15.1	7.8
Iowa	24.5	13.4	11.1

Casagrande[1], in discussing the accuracy of determinations using the liquid limit device, says:

> Increase in the colloidal content of soils tends to increase the difficulty of obtaining uniform mixtures. In such cases, the sides of the grooves were likely to contain streaks of soil whose true cohesion had not been entirely broken up. This condition appeared to be the cause of irregularities in the number of blows required to close the cut in successive trials, as well as a scattering of the points on the flow curve.

Again, in discussing the effect of the character of the soil on the accuracy of results:

> Difficulties were encountered in determining the flow curve of non-plastic soils largely because of insufficient adhesion to the cup and excessive permeability, the latter property causing excessive water content in the region of the groove.

It appears that there can be a variation of several percent in the consistency limits determined by standard procedure. The deviations between the limits determined by conventional methods and the method proposed here are, for the most part, within the deviations likely to occur using standard methods. It should therefore be possible to speed up the determination of the consistency limits without serious loss in accuracy by using the method described herein. This is especially valid for the estimation of the liquid limit.

Only a few *fat* clays were included among the soil samples used in this investigation. Indications are, however, that the moisture content corresponding to six inches of water pressure gives satisfactory results for the liquid limit. For the plastic limit, however, tensions greater than 1650 inches of water pressure appear to be required to give satisfactory results.

This study has covered only a part of the textural groups; others, such as clay loam, loam, and sandy loam, have not been investigated. Further research within the groups investigated as well as with a broader range of soil textural groups should be conducted to corroborate these findings.

CONCLUSIONS

1. If the textural classification of a soil is known, the consistency limits can be estimated by assuming them equal to the moisture content corresponding to a certain soil moisture tension.

2. Soil moisture tensions which give moisture contents approximating the consistency limits are as follows:

a. Liquid Limit:	
Silty loam and silty clay loam	—60 inches of water pressure
Silty clay	—15 inches of water pressure
Clay	— 6 inches of water pressure
b. Plastic Limit:	
Silty loam	— 168 inches of water pressure
Silty clay loam	— 415 inches of water pressure
Silty clay	— 913 inches of water pressure
Lean and medium clays	—1650 inches of water pressure

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FURTHER CORRELATION OF CONSISTENCY LIMITS OF LOWA LOESS WITH CLAY CONTENT

by

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(Iowa Academy of Science Proceedings, 64:407-412. 1957.)

INTRODUCTION

Mixing and manipulation of soil and water cause consistency changes at various moisture contents. These changes reveal important characteristics of the soil even though the condition of the soil-water system is not the same as that found in nature. The characteristics of consistency have been correlated with engineering behavior of soil. Together with mechanical analysis, these relationships provide a basis for the identification and engineering classification of soil and aid in judging its suitability for engineering structures.

Soil-water mixtures in various states are from a true liquid to a solid, depending on the amount of water in relation to the amount of soil. In 1911 the Swedish scientist, A. Atterberg, suggested two simple tests for determining the moisture content at the upper and lower limits of the moisture range in which a soil has the properties of a plastic solid. These tests for liquid limit and plastic limit are in use today. The numerical difference between these two limits is called the plasticity index of the soil, and its determination is essential in judging an engineering soil. The plasticity index measures the cohesive properties of a soil and indicates the degree of surface activity and bonding properties of the clay minerals present.

The Atterberg limits are three of the many routine engineering tests being used to study the engineering properties of Iowa loess. Previous work has shown a linear correlation between the Atterberg limits and clay content of soils from southwestern Iowa. In this research loess samples were studied from the entire state, which has been divided arbitrarily into five different areas to facilitate the study of the properties of the loess.

The 223 samples of Wisconsin age loess used in this correlation study were taken at various depths (figure 1). A-horizon samples have been excluded as well as samples containing more than 5 percent sand. A-horizon samples contained considerable organic matter, and the samples with more than 5 percent sand were from the basal portion of the loess sections. These

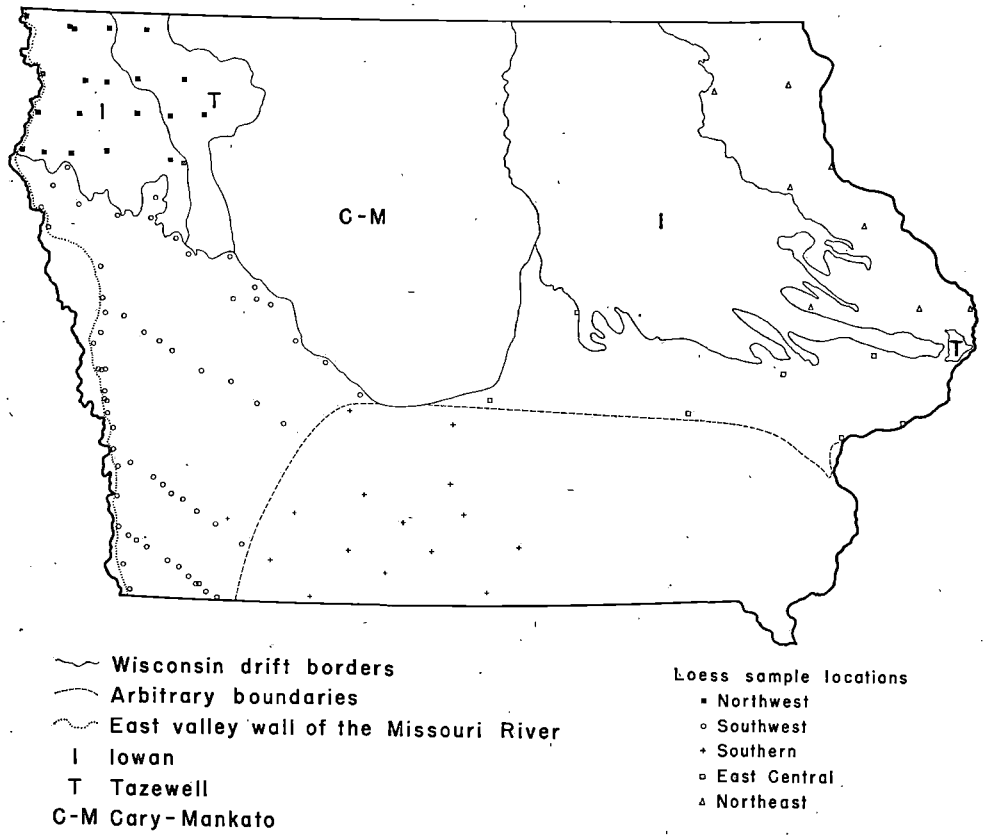

Fig. 1. Loess sample locations in Iowa.

samples tended to have erratic liquid and plastic limit values. Results from such samples were considered unreliable and were not plotted.

The correlations were made by plotting the Atterberg limits against the clay contents or all material with an equivalent spherical diameter of 5 microns or less (figures 2, 3, and 4). Linear correlations between the variables have been shown to hold for southwestern Iowa and were expected for the other areas. Subsequent plotting proved this true, and equations for the best straight line through the points from each area were determined by the method of least squares. Since the plasticity index is by definition the liquid limit minus the plastic limit, and the liquid limit and the plasticity index plots show the best relationships to clay content, the equations for the plastic limit plots were derived from those found for the liquid limit and the plasticity index (table I). The equation of a straight line is $y = mx + b$, where m is the slope of the line and b is the intercept. Here y represents the Atterberg limit and x represents the clay content.

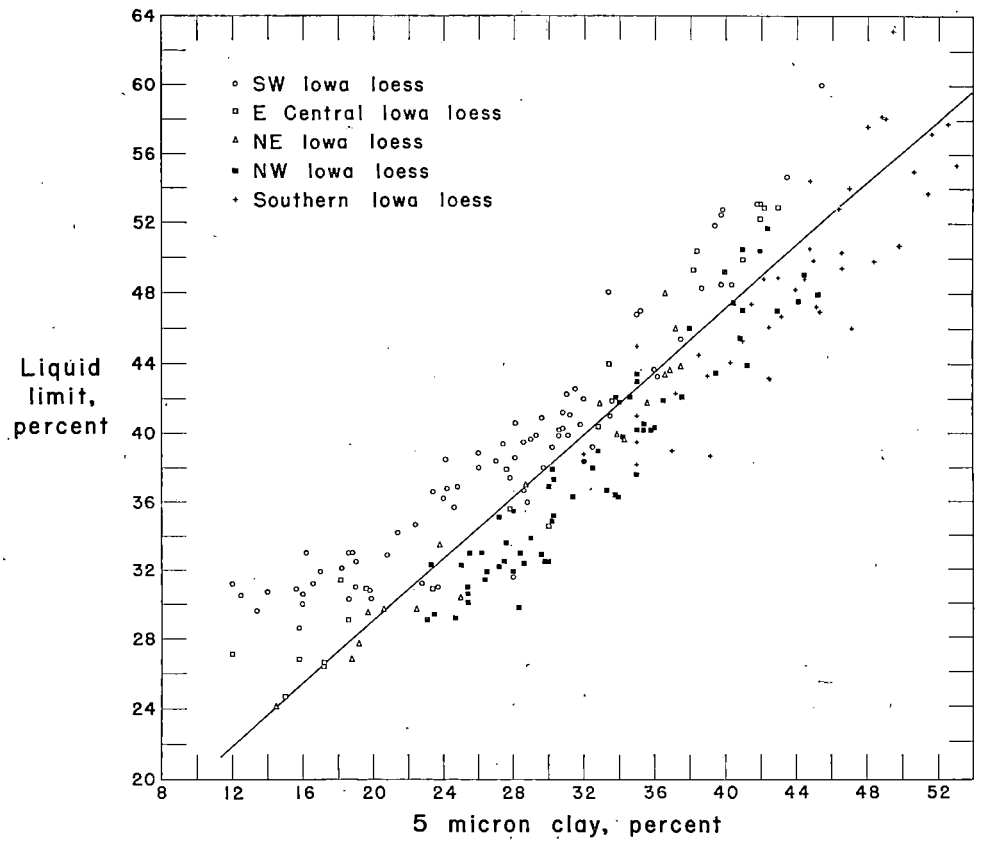

Fig. 2. Liquid limits plotted as a function of 5 micron clay content.

The liquid limit equations are all essentially parallel except for the one for southwest Iowa loess. This exception is believed to be due to a much higher carbonate content in the loess of this area, especially near the Missouri River where the clay content is low. Higher than normal liquid limits tend to reduce the slope of the line, but this is offset by the plastic limit equation. The resulting plasticity index equation exhibits a slope consistent with those of east-central and northeast Iowa.

The slopes of the plastic limit lines for northwest and southern Iowa loess are different from those found in the other three areas. The discrepancy was due to poor operator technique. All Atterberg limits for these two areas were run by the same operator. The liquid limit results can be duplicated easily by different operators, since it is mainly a mechanical test. The plastic limit requires judgment and it is therefore more subject to error.

In a comparison of plastic limits obtained by three different operators, the results of operator C agree quite well with those of operator A on the loess from southwest, east-central and northeast Iowa (table II). Operator

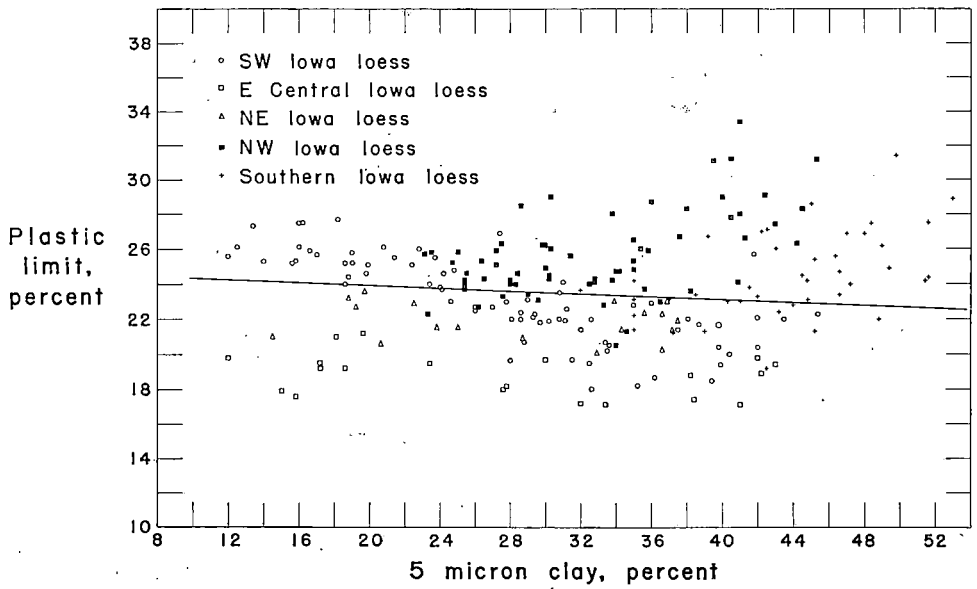

Fig. 3. Plastic limits plotted as a function of 5 micron clay content.

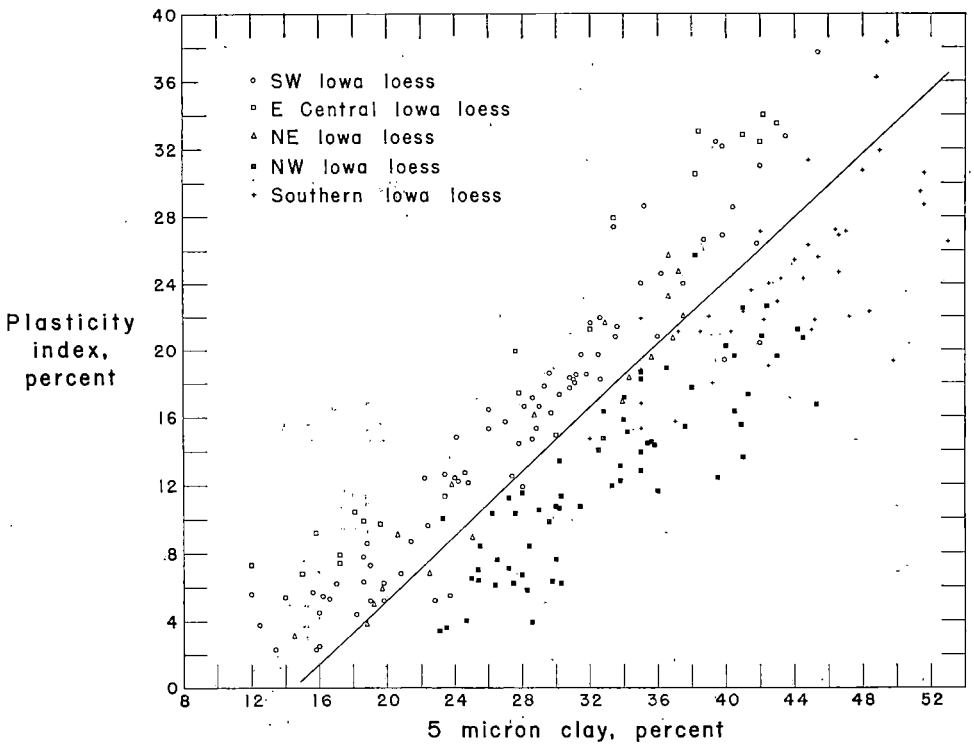

Fig. 4. Plasticity indices plotted as a function of 5 micron clay content.

TABLE I. COMPARISON OF CONSTANTS IN THE ATTERBERG LIMIT EQUATIONS

Atterberg limits = mx + b

Sample area	Liquid limit Slope m	Liquid limit Constant b	Plastic limit Slope m	Plastic limit Constant b	Plasticity index Slope m	Plasticity index Constant b
Southwest	0.79	16.9	—0.22	29.1	1.01	—12.2
East-central	0.95	11.3	—0.09	20.2	1.04	— 8.9
Northeast	0.93	10.0	—0.01	22.1	0.94	—12.1
Northwest	0.98	6.3	0.22	18.7	0.76	—12.4
Southern	0.99	5.2	0.25	13.2	0.74	— 8.0
Overall*	0.91	10.9	—0.04	24.7	0.95	—13.8

*Equations derived from lines drawn by eye through the entire mass of points.

B obtained results that appear to be much too high, especially in the higher clay content loess, for northeast and southern Iowa. From this comparison the plastic limit results of operator B are unreliable, probably due to insufficient training in the performance of this test.

TABLE II. COMPARISON OF PLASTIC LIMIT RESULTS OBTAINED BY DIFFERENT OPERATORS

Area	Sample No.	5-micron clay content, percent	P.L. by operators A and B, percent	P.L. by operator C, percent	Decrease A to C or B to C	Increase A to C or B to C
Southwest	55-1	13.4	A 27.3	C 23.8	3.5	
	20-2-VI	19.8	A 24.6	C 24.4	0.2	
	25-1	27.8	A 23.0	C 22.9	0.1	
	44½-1	42.0	A 20.4	C 21.1		0.7
East-central	122-10	17.2	A 19.5	C 19.1	0.4	
	110-6	23.4	A 19.5	C 19.6		0.1
	110-2	41.0	A 17.1	C 19.3		2.2
Northeast	227-5	14.5	A 21.0	C 20.5	0.5	
	222-4	28.7	A 20.9	C 19.5	1.4	
	202-2	34.3	A 21.4	C 19.7	1.7	
Northwest	300-8	25.4	B 23.7	C 23.3	0.4	
	300-4	31.4	B 25.6	C 23.4	2.2	
	308-3	37.6	B 26.7	C 22.7	4.0	
	305-2	45.3	B 31.2	C 24.3	6.9	
Southern	513-4	39.0	B 21.3	C 20.2	1.1	
	508-2	46.6	B 24.7	C 18.9	5.8	
	509-2	49.0	B 26.2	C 20.0	6.2	
	512-2	51.6	B 27.2	C 24.8	2.4	

A decrease in the plastic limit values that becomes larger with increasing clay content is indicated by the above comparison for the northwest and southern areas. Such a decrease would result in a flatter slope than is shown in Table I and bring the plastic limit and plasticity index results closer in agreement with the results from the other areas.

The straight line relationships of the Atterberg limits and the clay contents of the loess in the five areas of Iowa, together with the similarity of

the slopes of these lines, offer further evidence of the uniformity of the clay minerology of the loess. The correlations also indicate that differences in the Atterberg limits of the loess in Iowa are primarily due to the amount of clay present.

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PERMEABILITY TEST FOR SANDS

by

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(ASTM Symposium on Permeability of Soils. 1954.)

ABSTRACT

Information on the permeability of sand is often required in its utilization as an engineering material. A modification of Barber's falling head permeability test for porous granular materials is presented. The modified method is simple to use and gives reproducible results.

The modifications recommended include:

1) A procedure for loading and compacting the sample in the permeameter tube,

2) a carbon dioxide treatment to remove air from the sample, and

3) the repeated testing of the loaded sample until permeability values obtained are within a recommended range of variation.

The suggested permeability test was used to measure the permeability of several Iowa sands at various densities. Compositional information pertinent to the permeability of the sands is also presented.

DEVELOPMENT OF TEST METHOD

A falling head permeability test has been widely used for the laboratory determination of the permeability of sands[2]. This method is simple to use and does not require complicated equipment. However, test results obtained may not be readily reproducible mainly because of the lack of a specific procedure for loading the sample in the permeability tube. A modification of the test method, as given in the Appendix, is simple to use and gives reproducible results.

In developing the modified permeability test for sands, experiments were made for the following purposes:

1) To evaluate different methods of loading and compacting the sample in the permeameter tube,

2) to determine the effect of air entrapped in the loaded sample on permeability,

3) to determine the effect of soaking the loaded sample in water for varying periods of time prior to testing, and
4) to compare the permeability values obtained by repeated tests on the same loaded sample.

Loading and Compaction

In the permeability test, the loading is done by pouring an air-dried sample into a 2 inch diameter permeameter tube to a depth of 6 inches. Since the tube is usually 48 inches long, the pouring of dry sand in this manner may cause appreciable segregation of coarse and fine particles. Segregation can be avoided by loading the sample in a moist condition, but moist sand may be difficult to compact to a desired density.

A comparison was made of different techniques for loading dry sand in the permeameter tube, and the procedure, called the "inverted method", was found to be satisfactory. In this method, the permeameter tube is inverted and the sample is loaded in what is then the upper 6 in. of the tube.

TABLE I. COMPARISON OF REPRODUCIBILITY OF PERMEABILITY TEST RESULTS OBTAINED WITH SAMPLES* LOADED BY TWO DIFFERENT METHODS

Method of Loading	Experiment No.	Coefficient of Permeability, ft. per day
Inverted method	1	28.2
	2	29.2
	3	29.4
	4	30.9
Sample poured from top to bottom of permeameter tube	1	16.8
	2	25.3
	3	12.0
	4	19.6

*The data are for Sample 77-S in Table III.

After loading, the tube is turned right-side up for the permeability test. The details of the loading procedure are given in the Appendix. The typical test data illustrate the high degree of reproducibility in test results obtainable by the inverted method of loading (table I). The low reproducibility of results obtained with similar samples loaded by the method of pouring the sample from top to bottom of the permeameter tube are shown for comparison. All samples were tested for permeability according to Barber's procedure.

The density of a sample loaded by the inverted method is close to the compact unit weight[1]. Higher densities may be obtained by compacting the sample with a cylindrical weight as in the Appendix. Sands can be easily compacted to Standard Proctor density by this method.

Carbon Dioxide Treatment

The permeability test can be performed with the sample either in a completely saturated condition or in a partially saturated condition. The condition of testing usually depends on the purpose of the test. The simplest way to obtain results on a comparable basis is to use completely saturated samples. The permeability test for sands given in the Appendix was developed primarily for testing completely saturated samples. However, the procedure for loading and compaction may also be used for preparing partially saturated samples for testing.

To obtain complete saturation, the sample should be free from entrapped air before being tested for permeability, and no air should be introduced into the sample from the percolating water during testing. The entrainment of the air in the test sample can be prevented by evacuation methods[5] or by passing carbon dioxide gas through the air-dry sample after it has been loaded in the permeameter tube and before it is compacted. The air in the sample is displaced by CO_2 which will be quickly dissolved by percolating water during the permeability test. The latter method is comparatively simple to use and was found satisfactory in permeability tests with many types of soil[3]. It was the only method studied in the present investigation.

The two variables involved in the carbon dioxide treatment are the amount of CO_2 used and the rate at which it is passed through the sample. Experiments were performed with sand samples to determine a satisfactory amount of CO_2 used and the rate at which it is passed through the sample.

De-aired water may be used in permeability tests to avoid the release of dissolved air as the water percolates through the test sample. The use of distilled water at a temperature 5 to 10° F. higher than room temperature also serves to avoid the release of dissolved air. Because of its simplicity this method is recommended.

Experiments were made to determine the effect of air removal by the carbon dioxide treatment on the permeability of several sand samples. The samples were loaded in the permeameter tube by the inverted method and, by the method described in the Appendix, were compacted to various densities prior to permeability determinations[2]. Test data showed that the carbon dioxide treatment results in a higher permeability regardless of the type of sand and of the degree of compaction. The data also indicated that the lower the permeability of the untreated sample the greater the increase in permeability due to the carbon dioxide treatment (table II).

TABLE II. TYPICAL DATA SHOWING THE EFFECT OF CARBON DIOXIDE TREATMENT ON THE PERMEABILITY OF SANDS

Sample No.	Porosity, percent	Carbon Dioxide Treatment	Coefficient of Permeability†, ft. per day	Relative Permeability†, percent
77-S	39.6	No	29.4	76
		Yes	38.8	100
	36.6	No	12.0	49
		Yes	24.6	100
89-S	33.8	No	150.2	89
		Yes	168.7	100

*Properties of samples used are given in Table III.
†Data are the average of at least two tests.
‡Relative permeabilities are computed by taking the coefficient of permeability obtained with carbon dioxide treated samples as 100%.

Soaking

After the sample has been loaded in the permeameter tube, treated with carbon dioxide, and compacted to the desired density, the tube is immersed in distilled water in a water bath (Appendix). The permeability determination can be started either immediately after the water in the tube has risen to nearly the water level in the bath or after the sample has been soaked for an additional period of time. Experimental results indicate that additional soaking up to 48 hours does not appreciably change the permeability value. For this reason, a soaking period prior to the permeability determination is not considered necessary.

CHANGE IN PERMEABILITY DURING TESTING

In the falling head method of test, the permeability is determined on the basis of the time required for the water in the permeameter tube to drop from level A to level C (figure 7). Any change in the permeability of the sample during this time cannot be detected, since only one coefficient of permeability is computed. If an intermediate level, level B (figure 7), is marked on the permeameter tube and the time intervals required for the water to drop from level A to level B and from level B to level C are recorded, two coefficients of permeability can be computed. If this is done, any change in the permeability of a sample during testing is indicated by the difference between the two coefficients of permeability.

To facilitate the comparison of the two coefficients of permeability, an intermediate level can be selected to conform to the following relationship:

$$\frac{h_a}{h_b} = \frac{h_b}{h_c} \qquad (1)$$

Where:

h_a = height of level A,

h_b = height of the intermediate level (level B),

h_c = height of level C.

Equation (1) and the formula for computing the coefficient of permeability given in the Appendix show that if the permeability of the sample is constant during the test, the time intervals required for the water in the tube to drop from level *A* to level *B* and from level *B* to level *C* will be the same. For simplicity the time required for the water to drop from level *A* to level *B* will be referred to as the first reading, and the time from level *B* to level *C* the second reading. Any difference between the first and the second readings is an indication of a change in the permeability of the sample during testing.

Test results indicate that, when a single permeability determination is made on a sample, the first reading is usually different from the second. It was also found that the deviation between the two readings often decreases with repeated permeability determinations on the same loaded sample (figure 1). The deviation curve shows that after several successive

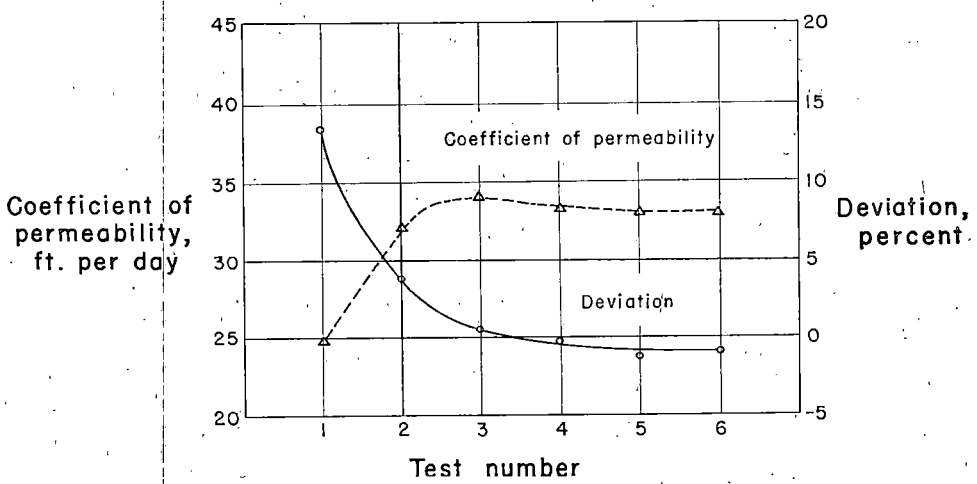

Fig. 1. Typical curves showing variation in permeability test results obtained by repeated testing of a sand sample (Sample No. 92-S, table III.)

permeability determinations the amount of deviation between the two readings becomes relatively small and remains nearly constant.

The permeability increases with repeated testing but approaches a uniform value as the deviation curve flattens (figure 1). The permeabilities obtained from tests having small deviations between first and second readings are, probably, more representative of the test sample than the permeabilities obtained from tests having large deviations. For this reason, it seems desirable in routine permeability tests to repeat the determination until the deviation between the first and second readings is small and, then, to report the coefficient of permeability obtained by this determination.

To determine the permissible deviation of first and second readings, an analysis of permeability and deviation data for several sand samples tested at various densities was made. As shown by the dotted lines, which show the relationship between permeability values and deviations, when the deviation is less than ±2.0 percent, the permeability value is within about ±5 percent (95 to 105 percent relative permeability) of the value corresponding to a deviation of nearly zero (figure 2). Since a 5 percent variation in

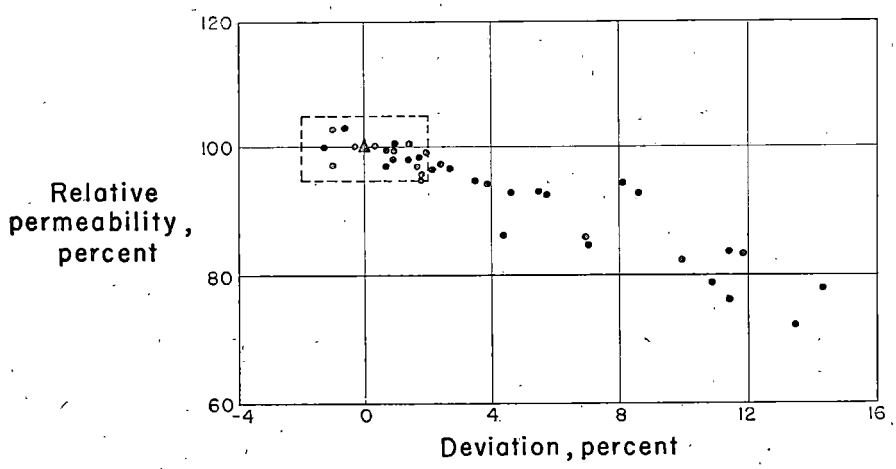

Fig. 2. Relationship between permeability value and deviation between first and second readings.

the permeability value seems to be a reasonable tolerance, ±2.0 percent is recommended as the allowable limit of deviation between first and second readings. In many tests, this requirement was met in the second permeability determination.

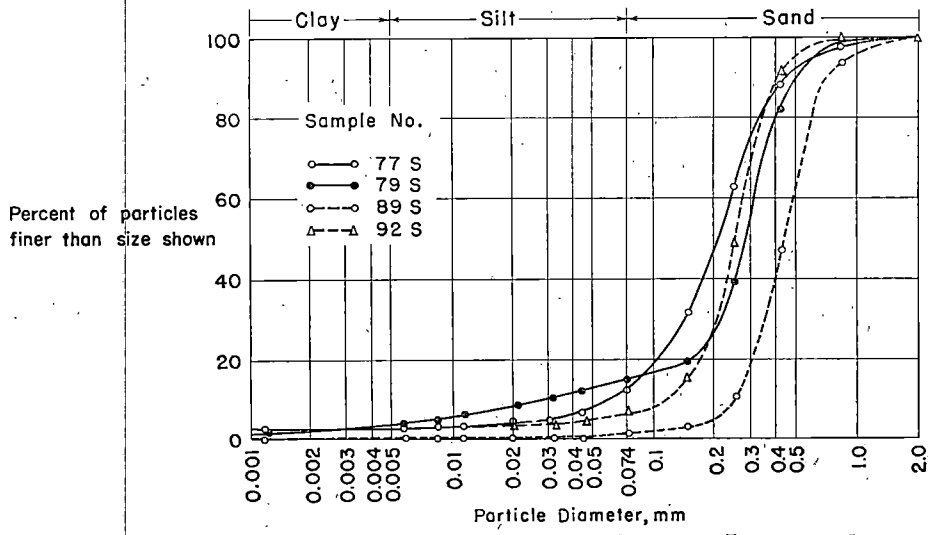

Fig. 3. Particle size accumulation curves for the four Iowa sands.

TABLE III. MECHANICAL COMPOSITION OF THE FOUR IOWA SANDS

Sample No.	Textural Composition* Sand, %	Silt, %	Clay, %	Sorting Coefficient	Sp. G.	Ave. Sphericity	Surface Texture† Markings	Coatings	Aggregation Characteristics Amount	Size, mm	Cement
77-S	87.6	9.6	2.8	1.6	2.66	0.71	Dull and rough (faceted)	Partially coated by calcareous clay	Common	1 - 3	Calcareous Clay
79-S	84.7	11.0	4.3	1.4	2.65	0.73	Dull and smooth	Partially coated by ferruginous clay	Abundant	½ - 1½	Ferruginous Clay
89-S	98.1	1.6	0.3	1.2	2.67	0.75	Dull and rough	Partially coated by iron oxide	Absent	.....	.....
92-S	92.9	4.3	2.8	1.3	2.68	0.61	Dull and rough (pitted)	Completely coated by slightly calcareous clay	Common	1 - 2	Slightly Calcareous clay

*Sand — 2 to 0.074 mm silt — 0.074 to 0.005 mm clay — Less than 0.005 mm.
†Descriptions apply to most sand-size particles in the sample.

PERMEABILITY OF FOUR IOWA SANDS

The modified test method was used in determining the permeability of four Iowa sands[7] (figure 3, table III). The sorting coefficient, S_o is computed by the formula[6]:

$$S_o = \sqrt{\frac{Q_a}{Q_b}}$$

The values of Q_a and Q_b are determined from the particle size accumulation curve: Q_a is the maximum diameter of the smallest 75 percent by weight of the soil particles, and Q_b is the maximum diameter of the smallest 25 percent. A well graded sand will have a higher sorting coefficient and will usu-

ally have a lower porosity than a poorly graded sand. The average sphericity values are for sand size particles in the sample[6]. A perfect sphere has a sphericity of 1.00. The predominant kind of clay mineral in each of the four sands is illite.

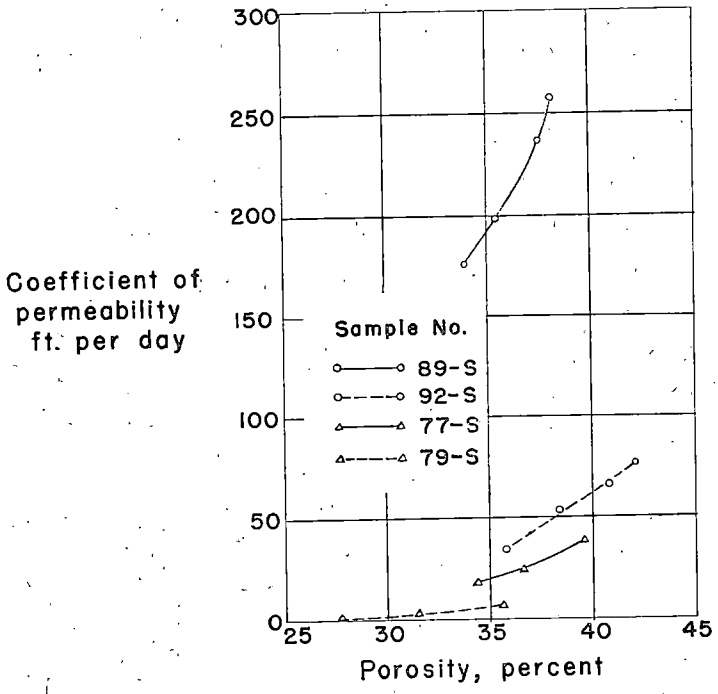

Fig. 4. Permeabilities of the four Iowa sands at various porosities.

TABLE IV. COEFFICIENTS OF PERMEABILITY OF FOUR IOWA SANDS AT VARIOUS DENSITIES

Sample No.	Dry Density: Lb. per cu. ft.	Dry Density: Percent of Standard Proctor Density	Porosity, percent	Coefficient of Permeability, ft. per day
77-S	100	92	39.6	39.5
	105	97	36.6	24.5
	109	100	34.5	18.4
79-S	106	90	35.6	6.9
	113	96	31.5	2.4
	119	101	27.8	1.4
89-S	103	96	38.1	258.5
	104	97	37.5	237.5
	108	100	35.5	199.4
	110	102	34.0	175.4
92-S	97	90	42.0	77.1
	99	92	40.8	66.3
	103	96	38.3	54.5
	107	100	35.7	34.1

The data for the permeability test results for the four sands at various densities show that the sands differ greatly in their coefficients of permeability and the coefficient varies with the density for each sand (figure 4, table IV).

The differences in permeability of the sands are related to their void characteristics. The term *void characteristics* as used here refers not only to the amount of voids in a sample but also to other related variables, such as the size distribution and continuity of the voids. The amount of voids in a sample can be easily determined and is commonly expressed in terms of the porosity of the sample. The other variables are difficult to measure quantitatively, but they can be evaluated qualitatively from compositional information.

Each curve in figure 4 shows the decrease in permeability that accompanies a decrease in porosity. The figure also illustrates that porosity is not the only important factor affecting permeability. For example, Sample 79-S at a porosity of 35 percent has a coefficient of permeability of about 6 ft. per day, but Sample 89-S tested at a similar porosity has a coefficient of about 200 ft. per day. This great difference in permeability is largely due to the differences in mechanical composition (figure 3, table III).

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APPENDIX

METHOD OF TEST FOR PERMEABILITY OF SANDS

Scope

This method of test determines the coefficient of permeability of sands. The term *sand* refers to the granular cohesionless material identified as *sand* in the Bureau of Public Roads textural classification system.

Apparatus

The apparatus used in this test consists of the following:

Permeameter Tube. A rigid transparent plastic tube approximately 48 in. in length and about 2 in. in inside diameter (figure 7).

Brass Screens. Two circular brass screens. One is of No. 200 sieve cloth, the other of No. 10 sieve cloth. The diameters of the screens are the same as the outside diameter of the permeameter tube.

Screen Holder. A holder to attach the screens tightly to the permeameter tube.

Supporting Device. A supporting device made of rubber stoppers, a brass tube, etc. (figure 5).

Loading Funnel. A special funnel (figure 5).

Tamping Rod. A straight $\frac{1}{8}$ in. round metal rod, approximately 8 in. long.

Attachment for Carbon Dioxide Treatment. An attachment made of a rubber sleeve, rubber stopper, etc. (figure 5).

Bottle of Carbon Dioxide and Flow Meter. A 50 pound tank of liquid carbon dioxide with a flow meter, accurate to 0.01 cu. ft. per min., attached.

Cylindrical Weight. A 4 pound steel or brass weight having a diameter of about $1\frac{7}{8}$ in.

Balance. A balance of 1 kg. capacity and accurate to 0.1 g.

Ruler. A ruler reading to $\frac{1}{32}$ in.

Thermometer. A thermometer accurate to 1 F. or 0.5 C.

Stop Watch. A stop watch reading to $\frac{1}{5}$ sec.

Water Bath. A water bath with attachments for holding permeameter tube (figure 7).

Loading of Test Sample

A representative air dry sample of sufficient quantity, usually 500 g., is prepared and its weight recorded. The sample is mixed until uniform. To obtain the oven dry weight of the sample, the air dry weight is corrected for hygroscopic moisture.

The sample is loaded in the permeameter tube in four layers, each approximately 1.5 in. thick. The loading funnel and supporting device (figure 5) is used in loading the sample by the following procedure:

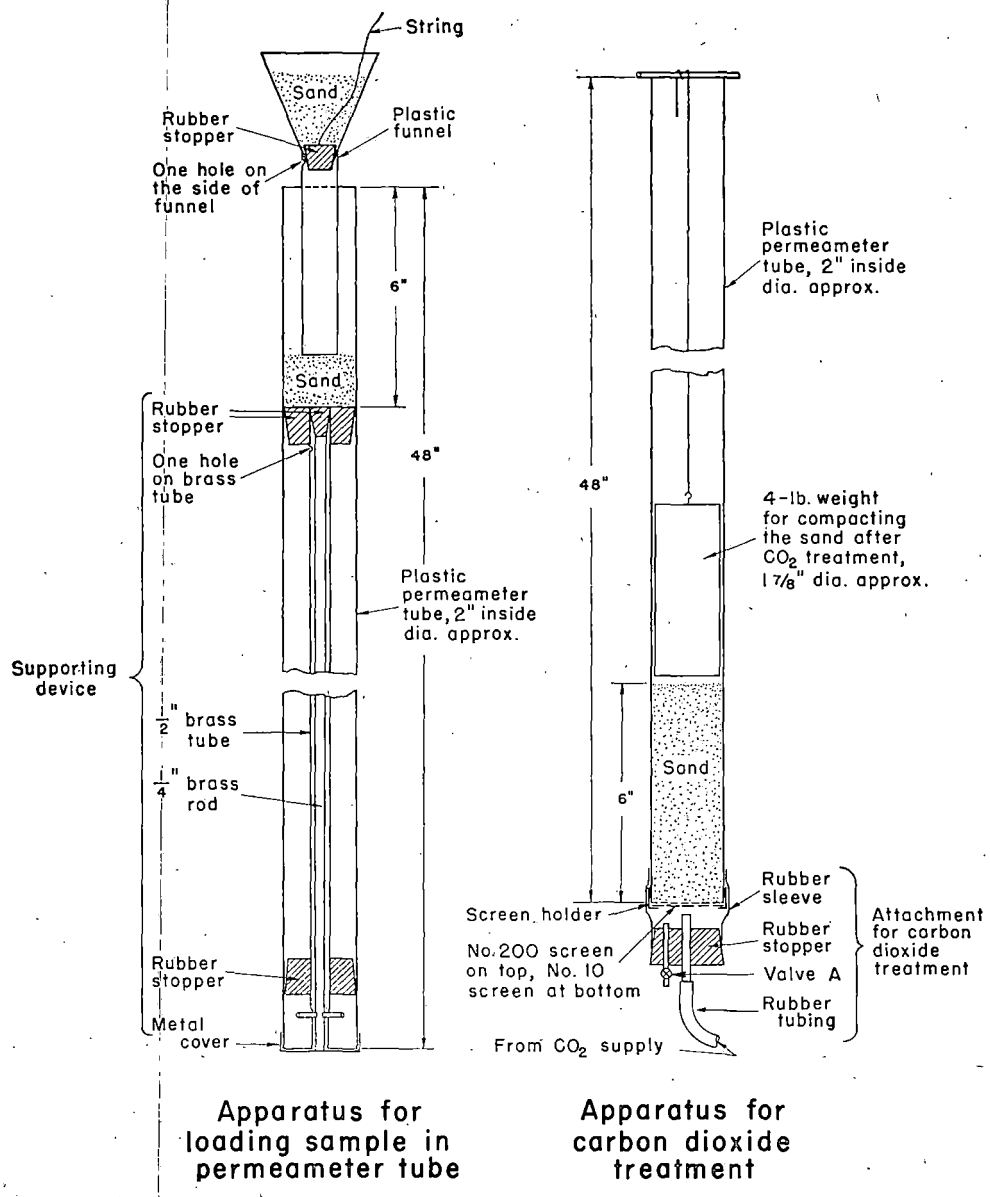

Fig. 5. Apparatus for preparing sample for permeability determination.

1) Invert the permeameter tube and assemble loading apparatus as shown in figure 5. For loading the first layer, the stem of the funnel rests on the top of the supporting device.

2) Pour a portion of the sample sufficient to make a layer approximately 1.5 in. thick into the stoppered funnel. If segregation occurs as the sample is being poured, remix to a uniform condition in the funnel.

3) Pull the stopper and allow the sand to flow down into the stem of the funnel. The small hole in the neck of the funnel permits the escape of air from the stem.

4) Carefully remove the funnel. The resulting layer will usually be free from segregation.

5) Use the tamping rod to rod the layer five times. The rodding is distributed uniformly over the layer and extends through it.

The above procedure is followed in loading each of the other three layers. After all four layers are loaded, any excess sand above the top of the permeameter tube is removed and saved for weighing. The weight of sand in the permeameter tube is determined by subtracting the weight of all excess sand from the initial weight of the sample.

Two brass screens are attached tightly to the top of the permeameter tube, the one of No. 200 sieve cloth being in direct contact with the loaded sample. The permeameter tube is then turned right-side up and the supporting device removed. In removing the supporting device, the following procedure is used:

1) Lift the rod inside the brass tube until the short cross bar is clear of the slots, and then rotate it about 90 degrees until the cross bar can be rested on the end of the brass tube. This step is necessary to prevent disturbance of the sample during removal of the supporting device.

2) With the permeameter tube in a vertical position, remove the whole supporting device slowly from it.

Carbon Dioxide Treatment

The sample in the permeameter tube is treated with carbon dioxide gas in the manner shown (figures 5, 6). The rate at which the carbon dioxide gas is passed through the sample is 0.03 cu. ft. per min. During the first ½ min. of the carbon dioxide treatment, valve *A* is left open to permit the escape of air from the chamber below the sample. The valve is then closed and the treatment is continued for an additional 15 min. The amount of carbon dioxide passed through the sample is about 0.45 cu. ft.

Compaction (Optional)

After the carbon dioxide treatment, the sample is compacted, if necessary, to the desired density before being tested for permeability. In compacting the sand, the 4 lb. cylindrical weight is placed on the sample in the permeameter tube. The portion of the tube containing the sample then is patted with the palm of the hand until the desired density is obtained.

Note: Since both the weight of the sample in the tube and the inside diameter of the tube are known, the depth to which the sample is compacted can be computed from the desired density.

Permeability Determination

The sample in the permeameter tube is immersed in distilled water at room temperature by one of the following procedures:

Method A: For samples loaded in the permeameter tube without additional compaction (figure 7). After the carbon dioxide treatment, the sample is gently immersed in the water bath.

Method B: For samples loaded and further compacted in the permeameter tube (figure 7). The sample is very gently immersed in the water bath with the weight used for compaction on top of the sample to prevent expansion during immersion. The weight is removed when the rising water nears the top of the sample.

After the water in the permeameter tube has risen to the level of the water bath, the tube is filled with distilled water, at a temperature between 5 and 10° F. above room temperature, without disturbing the sample in the

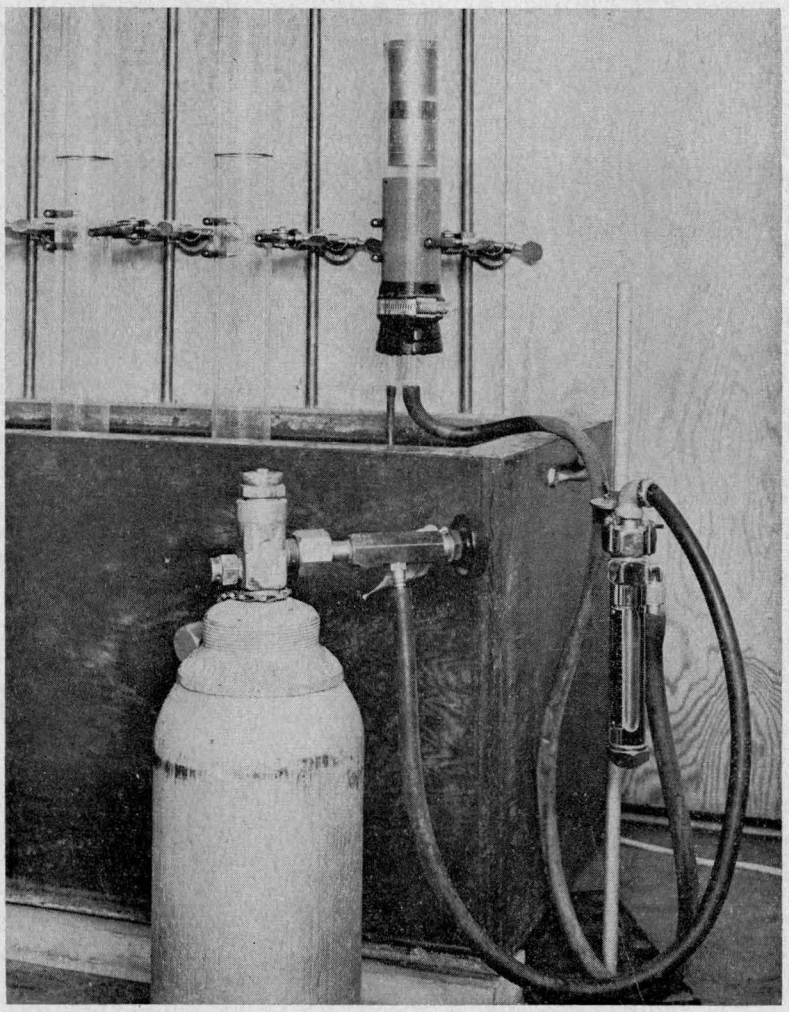

Fig. 6. Carbon dioxide treatment of sand samples.

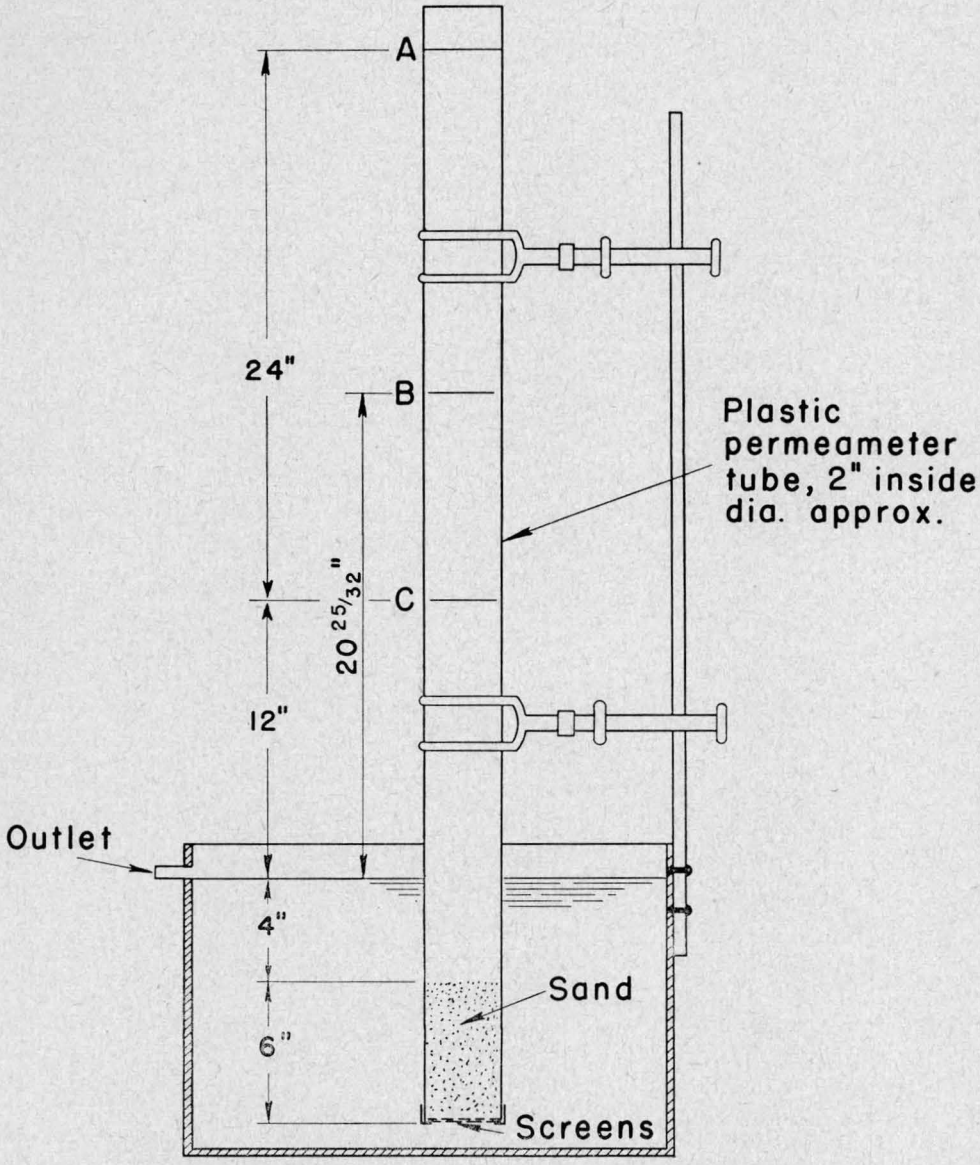

Fig. 7. Arrangement of apparatus for permeability determination.

tube. The time intervals required for the water in the tube to drop from level *A* to level *B* and from level *B* to level *C* are recorded with a stop watch.

If the difference between the first and second time intervals is more than 2.0 percent of the average value, the preceding step is repeated one or more times until the difference is less than 2.0 percent. The depth and the tem-

perature of the sample in the permeameter tube is then measured.

Note: Fig. 8 illustrates the assembly of apparatus for testing a group of samples simultaneously.

Calculation

The coefficient of permeability, k, in ft. per day, is calculated as follows:

$$k = \frac{276\ Cd}{t} \log \frac{h_a}{h_c}$$

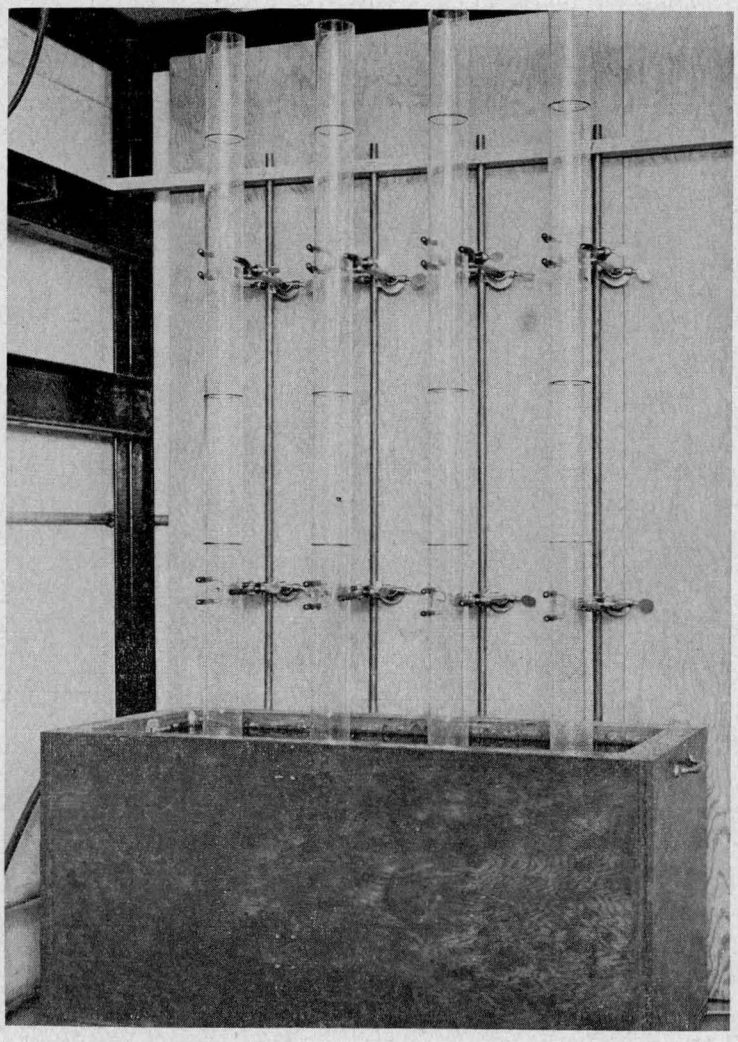

Fig. 8. Arrangement of apparatus for testing a group of samples.

Where:

C = temperature correction (viscosity of water at the temperature measured in the sample after testing divided by viscosity of water at 68° F.),

d = depth of sample in inches measured after the test,

h_a = height of level A from the water level in the bath,

h_c = height of level C from the water level in the bath,

t = time in minutes required for water to drop from level A to level C during the last permeability determination.

NOTE: For $h_a = 36$ in. and $h_c = 12$ in., the above formula can be simplified to:

$$k = \frac{131.7\ Cd}{t}$$

SOME LABORATORY TESTS FOR THE EVALUATION OF STABILIZED SOILS

by

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D. T. Davidson, Professor, Civil Engineering

(Progress Report, 1955.)

ABSTRACT

The test methods presented in this paper include a test for determining the moisture-density relationships of natural or stabilized soils; a flexural test; an unconfined compression test; and the Miniature Bearing Test. Soil specimens used for the four tests are cylindrical in shape and two inches in diameter. The value of the small specimens is in the saving of time and material in making evaluation tests of stabilized soils.

The apparatus and procedure for each test given in the paper are for the testing of silty and clayey soils. However, it is believed that, with some modifications, the test methods may also be applied to the testing of sandy soils.

INTRODUCTION

Various kinds of organic and inorganic materials have been investigated recently for use as soil stabilizing agents in the construction of highways and airports. Since the properties and environmental conditions of soil vary so greatly from place to place, a stabilizing agent which is suitable for one type of soil may not be satisfactory for another type. Because of this, it is often desirable to evaluate several stabilizing agents before deciding on a specific one to be used for a given type of soil. Tests for exploratory or preliminary evaluations for some soils are required for screening a comparatively large number of stabilizing agents; the most promising ones may then be selected for further investigations. Exploratory or preliminary evaluation tests are also useful in research work related to soil stabilization.

Laboratory tests for the purposes discussed above should be simple in procedure and should be applicable for the testing of soil stabilized with various kinds of additives. Four test methods were developed to meet such requirements. In the past four years, the test methods have been satisfac-

torily used in soil stabilization studies conducted by the Iowa Engineering Experiment Station.

In developing the test methods, experiments were made mostly with silty and clayey soils, and the test procedures given in this paper are for these two types of soil. However, it is believed that, with some modifications in the test procedures, the test methods are also applicable for the testing of sandy soils.

DENSITY TEST

One of the common tests with stabilized soils is the determination of their moisture-density relationships. The standard or the modified Proctor density test[1, 4] using cylindrical specimens 4 in. diameter by 4.59 in. high may be used for this purpose. However, a test using smaller specimens is desirable for fine grained soils because of the savings in time and material

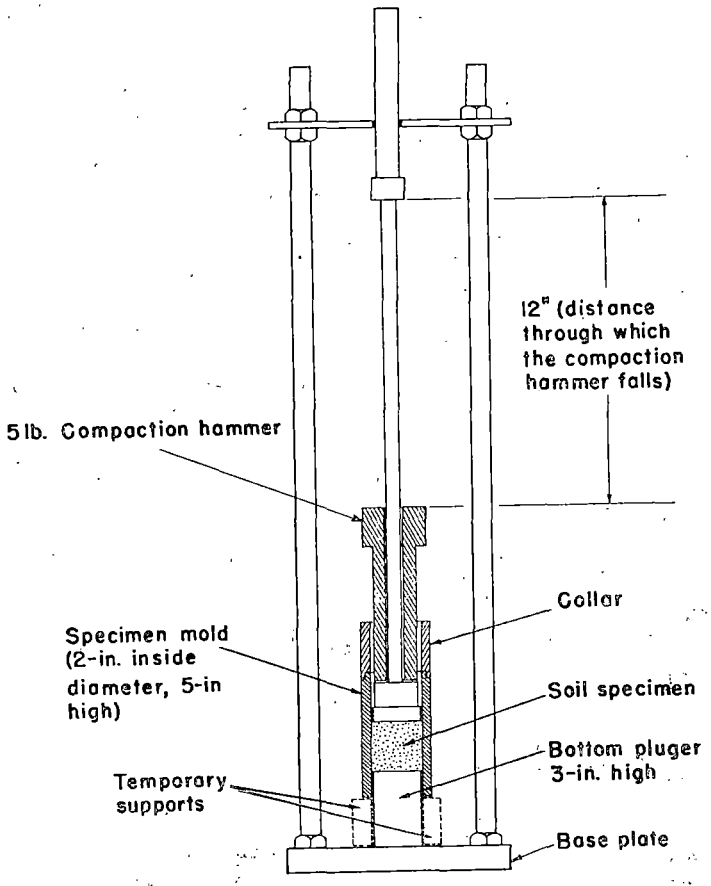

Fig. 1. Apparatus for molding 2 in. diameter by 2 in. high soil specimens.

needed for molding soil specimens. In the soil stabilization studies conducted by the Iowa Engineering Experiment Station during the past four years, a density test employing 2 in. diameter by 2 in. high specimens has been used satisfactorily for the testing of silty and clayey soils (figures 1, 2). A 5 pound hammer is dropped from a height of 12 in. to furnish the compactive effort. The procedure for performing the test is as follows:

1. Prepare a soil sample at desired moisture content.
2. Weigh out enough moist soil to make a specimen slightly over the 2 in. height after compaction.
3. Pour the moist soil into the specimen mold which rests on two temporary supports (figure 1).
4. Give one blow to the soil in the mold, remove the temporary supports, and give another four blows.

Fig. 2. Molding 2 in. diameter by 2 in. high soil specimens.

5. Remove the collar and invert the mold. The top of the partially compacted specimen in the mold becomes the bottom and is in contact with the bottom plunger.

6. Complete the compaction by giving five more blows to the specimen.

7. With the compacted specimen in the mold and resting on the bottom plunger, push the mold downward until it is in contact with the base plate. Since the mold is 5 in. high and the bottom plunger 3 in. high, the portion of the specimen in excess of 2 in. in height is pushed out of the mold. Trim off the excess soil to make the specimen exactly 2 in. high.

8. Weigh the specimen and take a sample for moisture determination. The dry density of the specimen can then be computed.

9. Repeat the procedure given above as the moisture content of the soil is increased.

A comparison of the maximum dry densities and the optimum moisture contents determined by the above test and by the standard Proctor density test with several raw and stabilized soils is shown (table II). The source and

TABLE I. SOURCE AND PROPERTIES OF SOIL SAMPLES

Soil		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
Source		Iowa	Texas	Virginia	Kentucky	Iowa	Iowa
Soil series		Hamburg	Lake Charles	Davidson	Melvin	Marshall	Webster
Horizon		C	C	B	C	C	A
Engineering classification (AASHO)		A-4(8)	A-7-6(20)	A-7-5(18)	A-6(8)	A-7-6(18)	A-7-5(15)
Textural composition,* percent	Sand	0.7	7.7	3.4	7.7	0.4	18.9
	Silt	78.3	48.2	12.0	55.7	60.2	37.1
	Clay	21.0	44.1	84.6	36.6	39.4	44.0
	Colloids	15.8	36.8	72.9	19.4	29.8	28.0
Liquid limit, percent		32	57	75	33	52	54
Plasticity index		7	37	24	11	33	20
Shrinkage limit, percent		25.2	14.4	27.3	22.9	19.1	20.7
Centrifuge moisture equivalent, per cent		15.2	21.2	29.5	21.4	28.5	25.7
Cation exchange cap., m.e./100g.		13.4	25.5	11.3	11.1	24.4	40.9
pH		7.8	5.9	4.1	4.5	6.7	7.4
Carbonates		10.2	2.7	1.0	1.2	0.5	4.7
Organic matter, percent		0.2	0.6	0.5	0.9	0.4	10.0

*Based on definitions of sand, silt, clay, and colloids given in ASTM test method D422-54T.

properties of the soils are given (table I). Note that for each soil, the maximum dry densities obtained by the two tests are nearly the same and the optimum moisture contents obtained differ only slightly. The moisture-density relationship of soil No. 1, a silty soil, and soil No. 2, a clayey soil, are illustrated in figures 3 and 4. For each soil, the moisture-density curves determined by the two tests are similar in shape. The same holds true for the other raw or stabilized soils represented in table II.

TABLE II. COMPARISON OF TEST RESULTS OBTAINED BY THE RECOMMENDED DENSITY TEST AND THE STANDARD PROCTOR DENSITY TEST

Soil	Additive		Recommended density test		Standard Proctor density test	
	Kind	Amount (percent of dry soil)	Max. dry density, lb. per cu. ft.	Opt. moisture content, percent	Max. dry density, lb. per cu. ft.	Opt. moisture content, percent
No. 1	Lime-fly ash (1:1)	33	97.5	20.3	96.5	21.3
	Aniline-furfural (2:1)	5	103.8	17.0	105.4	16.5
	No		109.9	18.2	108.7	18.0
No. 2	No		109.2	18.9	108.3	19.2
No. 3	No		85.6	37.1	83.2	37.0
No. 4	No		108.6	17.7	108.2	18.1
No. 5	No		100.5	20.2	102.0	20.7
No. 6	No		86.9	27.9	85.3	26.4

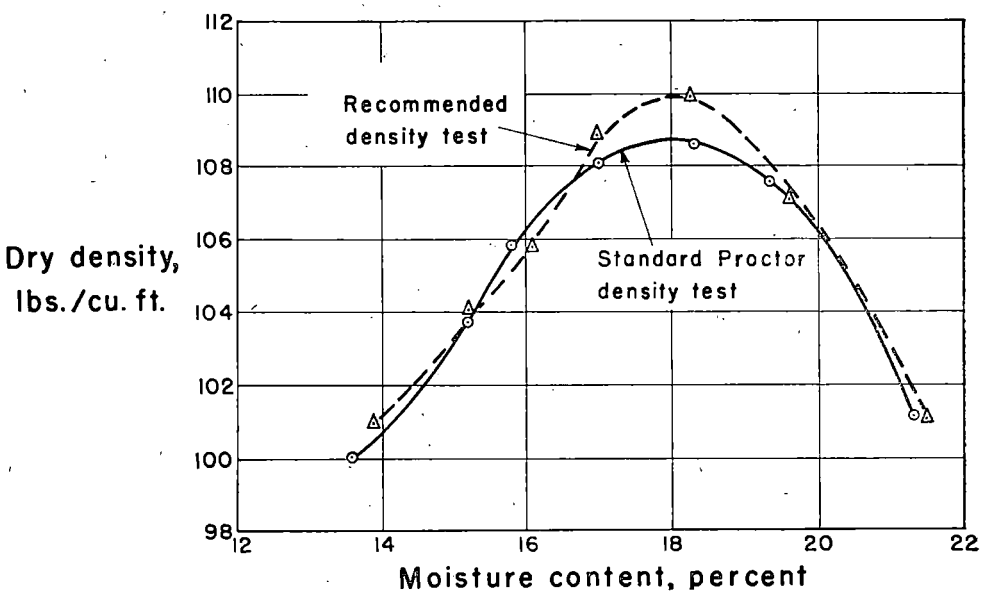

Fig. 3. Comparison of moisture density relationships for a silty soil, soil No. 1, determined by the recommended test and the standard Proctor density test.

Although a compactive effort of five blows of the hammer on each side of a specimen has been found satisfactory in all experiments to date, a verification of this compactive effort is desirable for soils having properties significantly different from those shown. If necessary, the number of blows may be so modified that results of the standard Proctor density test can be closely duplicated. Similarly by varying the number of blows, the test may be used for determining the moisture-density relationships of sandy soils.

The density test discussed above requires only about one-tenth of the material and one-third of the time needed for performing the standard

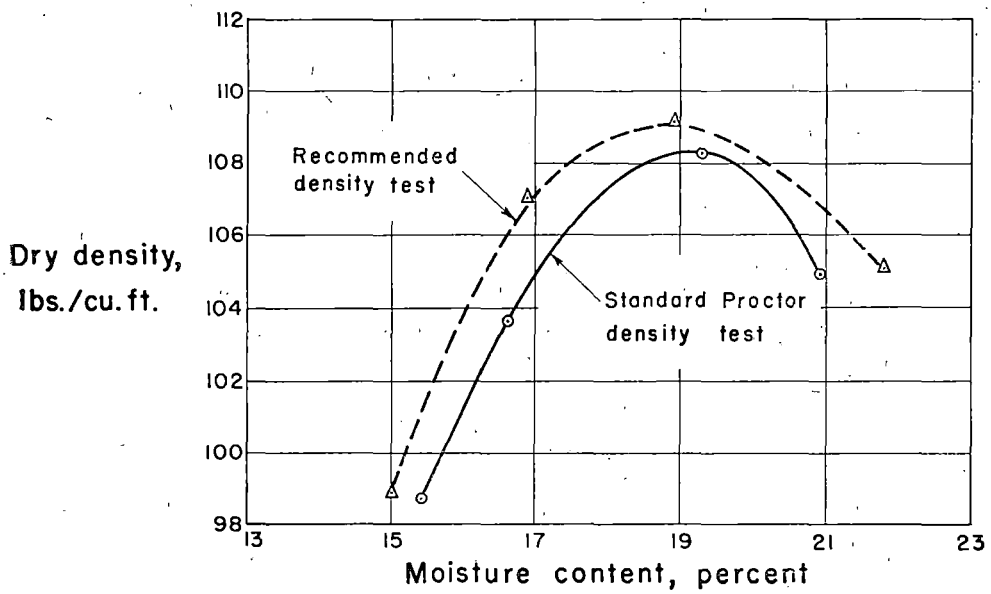

Fig. 4. Comparison of moisture density relationships for a clayey soil, soil No. 2, determined by the recommended test and the standard Proctor density test.

Proctor density test. The data from experiments performed indicate that results obtained by the above test are more reproducible than those obtained by the standard Proctor density test. It is believed that results of the modified Proctor density test can also be duplicated by a similar procedure except that the weight of the compaction hammer, the number of blows, or the height of the drop should be modified to obtain the necessary increase in compactive effort.

The suggested test is not intended to supplant the standard Proctor test for all moisture-density determinations in connection with the design and control of earth work or pavement construction. The principle use of the test is in the evaluation of soil stabilizing agents. For example, lime and fly ash are to be investigated as stabilizing agents for a silty or clayey soil. Since the addition of lime and fly ash to a soil will usually alter the moisture-density relationship of the soil, it is desirable to determine the maximum dry densities and optimum moisture contents of several mixtures containing various proportions of lime, fly ash, and the soil. The use of the suggested test for such purposes, instead of the standard Proctor density test, will result in savings in time and materials.

FLEXURAL TEST

The flexural test, which is simple in procedure and requires only a small quantity of material, is suitable for exploratory studies of various stabiliza-

tion methods. Those methods which appear to be promising may be further investigated by other types of tests. The flexural test is useful also for research in soil stabilization. For example, it is sometimes necessary to prepare test specimens from specially treated soils such as those having sodium or calcium as the predominant type of adsorbed cation. Since much time and expense are required for preparing large amounts of such treated soils, the use of small specimens is very desirable.

Soil specimens used for the flexural test are 2 inches in diameter and a half inch thick. Specimens of this size may be prepared by using the compaction equipment shown (figures 1, 2), except that the compaction hammer falls only 3 inches. The procedure for molding flexural test specimens is as follows:

1. Weigh out sufficient sample to make a specimen of a half inch ± 0.02 in height.
2. Pour the sample into the specimen mold. The collar is not needed in molding specimens for this test.
3. While the specimen mold is resting on the two temporary supports, give one blow of the hammer to compact the material in the mold.
4. Remove the two temporary supports and give another nine blows.
5. Extrude the specimen, then weigh it and measure its height.

For the silty and clayey soils used in the experiments, the density of flexural test specimens prepared in this manner is very close to that obtainable by the use of the standard Proctor density test.

Specimens prepared may be cured by any of several methods before being used for the flexural test. For example, soil-cement specimens are usually cured for seven days in a moist cabinet at a temperature near 70° F. and a relative humidity of not less than 90 percent. To simulate the effects of adverse weather conditions, specimens may be immersed in water for 24 hours before being tested for flexural strength.

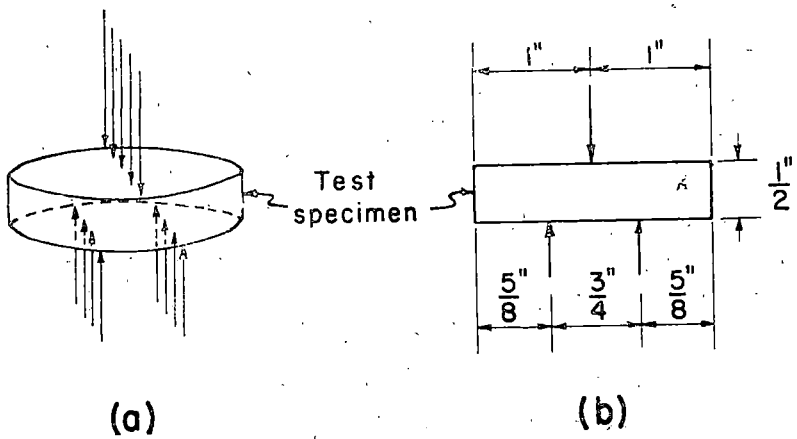

Fig. 5. Diagram showing the application of load in the flexural test. (a) Perspective of a test specimen. (b) cross-section through the center of the specimen.

The flexural test of soil specimens is made in the manner shown in figure 5, and an apparatus which may be used for the test is shown in figure 6. The test loading may be either by the use of an ordinary testing machine or by a direct shear test apparatus capable of registering loads to the nearest pound. The rate at which the load is applied should be one-tenth inch per minute; the maximum load causing failure is taken as the flexural strength of the specimen.

Although the modulus of rupture of a material tested may be computed from its flexural strength, it appears satisfactory to report only the flexural

TABLE III. FLEXURAL STRENGTH OF SOIL NO. 1* STABILIZED WITH VARIOUS ADDITIVES

Additive		Method of curing	Flexural strength, lb. (after 24-hr. immersion)
Type	Amount (percent of dry soil)		
Portland cement, Type I	10	7 days in moist cabinet at approximately 70° F.	49
Hydrated lime	3	Same as above	7
Lime-fly ash (1:3)	22	Same as above	17
Raw soil with no additive		No curing	Slaked during immersion

*See table I for properties of the soil.

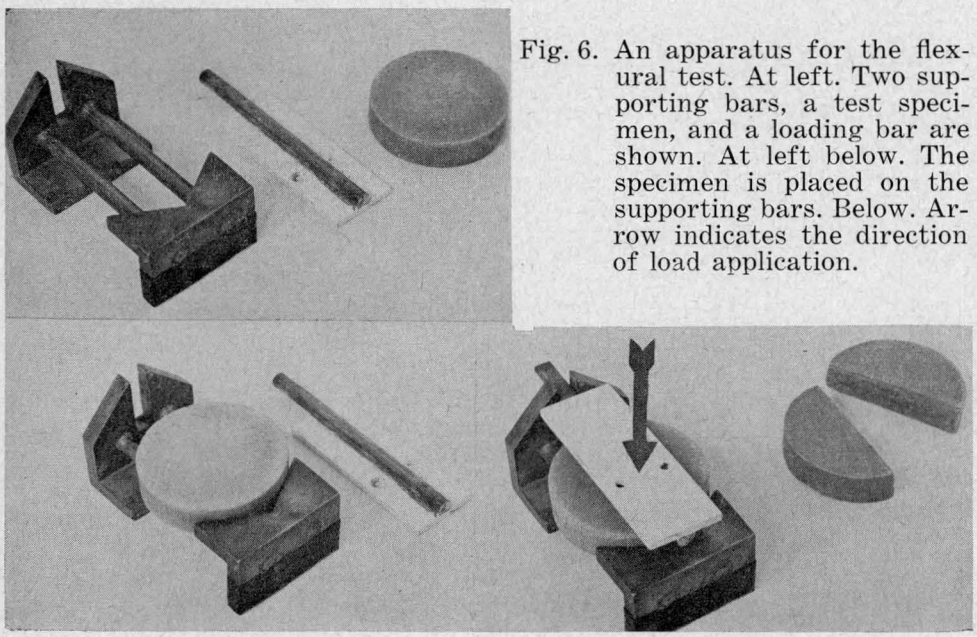

Fig. 6. An apparatus for the flexural test. At left. Two supporting bars, a test specimen, and a loading bar are shown. At left below. The specimen is placed on the supporting bars. Below. Arrow indicates the direction of load application.

strength values in using the test for preliminary evaluations of stabilized soils. The test data of a soil stabilized with various additives are shown in table III. The effectiveness of the additives is indicated by the flexural strength values given in the table.

In addition to the flexural strength, the water absorption and volume change of test specimens during the 24 hour immersion period may be useful for the evaluation of stabilized soils. Such information can be obtained by weighing and measuring the specimens before and after immersion.

UNCONFINED COMPRESSION TEST

The unconfined compression test, which is also simple in procedure and requires no special equipment, has been used by many investigators in the study of stabilized soils. Soil specimens of the size employed in the density test may be used for this test. If stabilized soils at a density obtainable by the standard Proctor density test are to be evaluated, test specimens may be prepared according to the procedure given for the density test except that the trimming of a specimen to exactly 2 inches in height may be omitted. Specimens two inches $\pm$ 0.05 inch in height were satisfactory for the unconfined compression test.

Specimens having a height to diameter ratio of two or more are desirable for compressive strength tests. The use of 2 inch by 2 inch specimens, with a height to diameter ratio of one, is mainly for convenience in preparing the specimens. Unconfined compression tests may also be performed by using cylindrical specimens having a convex or concave shaped top and bottom[2]. The strength characteristics of such specimens differ from that of cylindrical specimens with flat top and flat bottom. For preliminary evaluations of stabilized soils, cylindrical specimens with flat top and flat bottom appear to be satisfactory.

Specimens of stabilized soils may be cured by any of several methods as discussed previously. Cured specimens may be used for the unconfined compression test with or without further treatments. Common methods of treatment are immersion in water, wetting and drying, and freezing and thawing[6, 7]. The simple procedure of immersing specimens in water for 24 hours before testing was found satisfactory for preliminary evaluation purposes.

The unconfined compression test is performed at a rate of deformation of one-tenth inch per minute; the maximum load causing failure of the specimen is taken as its compressive strength. Although the strength of a specimen in pounds per square inch may be computed from the test data, it is convenient to report only the compressive strength in pounds. Data of the unconfined compression test with a soil stabilized with several kinds of additives are given in table IV. The effectiveness of the additives tested

TABLE IV. UNCONFINED COMPRESSIVE STRENGTH OF SOIL NO. 1*
STABILIZED WITH VARIOUS ADDITIVES

Additive Type	Amount (percent of dry soil)	Method of curing	Compressive strength, lb. (after 24 hr. immersion)
Portland cement, Type I	15	7 days in moist cabinet at approximately 70° F.	1780
Hydrated lime	6	Same as above	250
Lime-fly ash (1:2)	18	Same as above	485
Aniline-Furfural (2:1)	5	7 days air drying	1100
Raw soil with no additive		No curing	Slaked during immersion

*See table I for properties of the soil.

is indicated by the compressive strength values shown in the table. Compressive strength requirements of Portland cement stabilized soils have been studied by previous investigators[5, 8]; more studies are needed before specific values can be recommended as minimum requirements for all types of stabilized soils.

Similar to the flexural test, the water absorption and volume change of test specimens during the 24 hour immersion period may also be determined. Such information is often helpful in evaluating various methods of soil stabilization.

MINIATURE BEARING TEST

The California Bearing Ratio Test[3, 9] using soil specimens 6 inches in diameter and 5 inches high, has been widely used for evaluating the supporting strength of materials to be used in subgrades, subbases, or base courses for highway and airport pavements. The main objection to the use of this test is the preliminary evaluations of stabilized soils appears to be the large amount of material and the comparatively long time needed for preparing the 6 inch by 5 inch specimens. To get around this objection, attempts were made to use relatively small specimens in a test of similar nature. For silty and clayey soils, specimens 2 inches in diameter and 2 inches high were found satisfactory. The test using specimens of this size is called the Miniature Bearing Test.

The Miniature Bearing Test may be used for testing either raw or stabilized soils. The 2 inch by 2 inch test specimen can be prepared by using the equipment shown (figures 1 and 2). If a raw or a stabilized soil at standard Proctor density is to be evaluated, specimens may be prepared according to

the procedure given for the density test discussed previously. The specimen prepared is left in the mold and is not extruded throughout the test. If curing is desirable, the specimen is cured in the mold.

The bearing test may be performed with specimens which have or have not been immersed in water. If a specimen is to be immersed, the immersion can be done with or without an annular weight above the specimen. In testing raw soils, it is desirable to use an annular weight similar to that used in the California Bearing Ratio Test[3, 9]. When the Miniature Bearing Test is used for preliminary evaluation of stabilized soils, the annular weight may be omitted to simplify the test procedure.

The specimen without annular weight on it should be immersed long enough for it to absorb as much moisture as it can in a reasonable period of time (figure 7). The rate of moisture absorption depends on the type of soil and the stabilizing agent used. The water absorption during immersion may be determined by weighing the specimen together with the mold before and after immersion. The expansion of a specimen during the immersion period may also be determined. This can be done by clamping the mold to a perforated base and measuring the expansion in a manner similar to that of the California Bearing Ratio Test[3, 9].

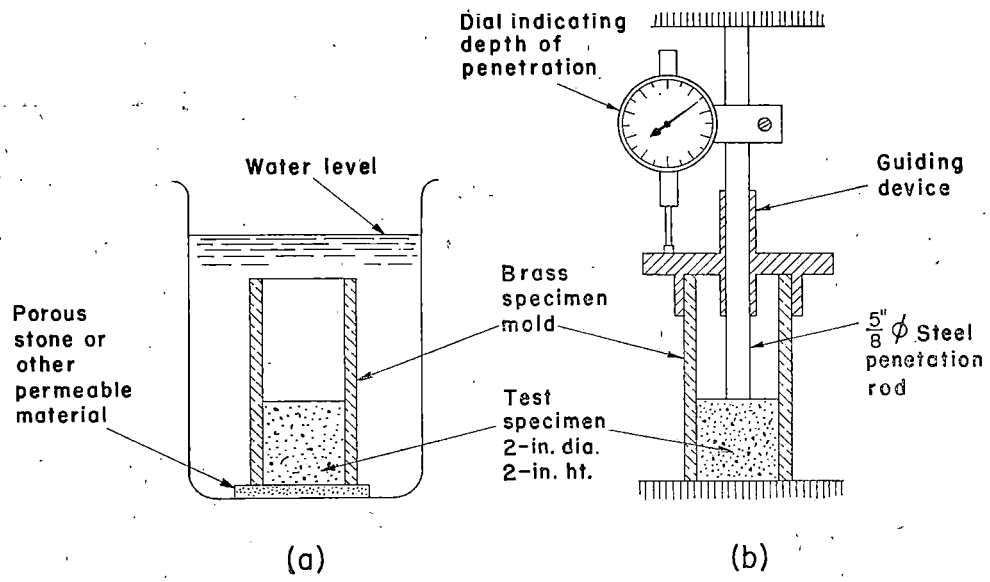

Fig. 7. The miniature bearing test apparatus. (a) Immersion of a test specimen in water bath. (b) Specimen in position for testing.

Before an immersed specimen is tested for bearing strength, it is removed from the water bath and allowed to drain for five minutes. During draining, the specimen is placed in an inverted position, i.e., the bottom of

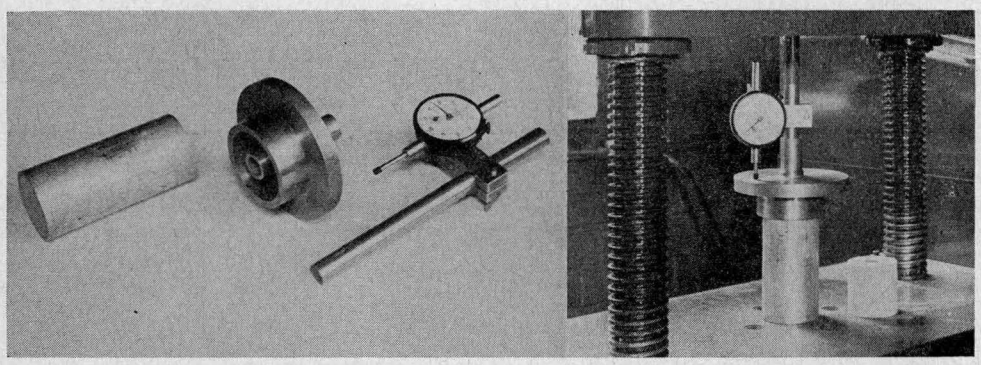

Fig. 8 Apparatus for the miniature bearing test. At left, the apparatus is disassembled. At right, the apparatus is assembled and placed on the compression table of a testing machine. A specimen which has been tested and extruded from the mold is shown to the right of the apparatus.

the specimen during immersion is kept upward. The bearing test is performed by using the apparatus shown (figures 7 and 8). In placing the penetration rod on a specimen, care should be taken not to disturb the surface of the specimen. The test load, which may be furnished by an ordinary testing machine, should be so applied that the rate of penetration is five hundredths inch per minute. The depth of penetration during testing is indicated by the dial attached to the penetration rod.

The relationship between the test load and the depth of penetration is illustrated in figure 9. The data shown were obtained by testing an uncured soil-bituminous specimen after 7 days immersion in water. It may be noted

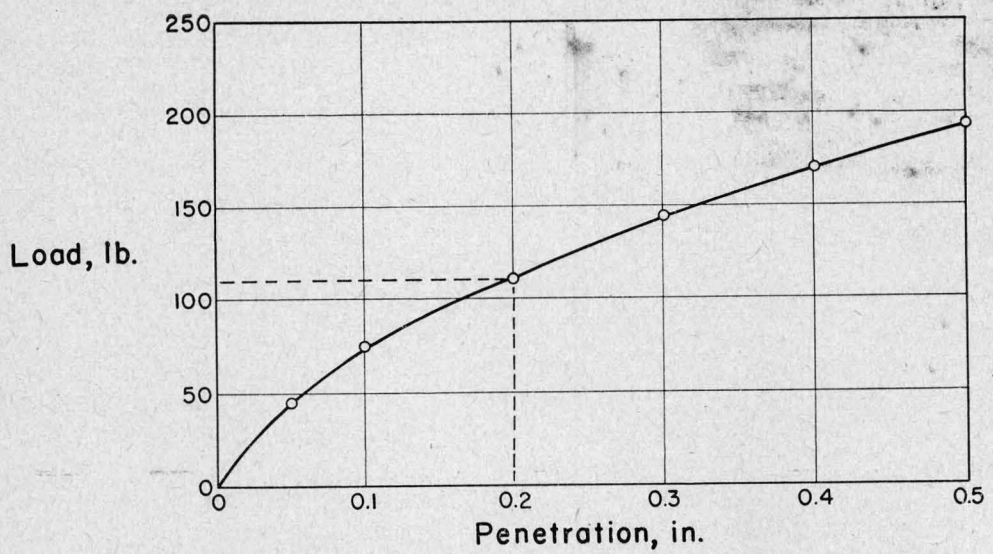

Fig. 9. Load-penetration curve of soil No. 1 stabilized with eight percent of cutback asphalt (MC-2). The miniature bearing strength of the stabilized soil is 110 lb.

that the test load increased continuously with the depth of penetration. The bearing strength of soils tested may, therefore, be evaluated on the basis of the test load at various depths of penetration. A tentative procedure for reporting the total load at two-tenths inch penetration was found satisfactory when the test was used for preliminary evaluation of stabilized soils. This load is termed the miniature bearing strength. The miniature bearing strength of the soil-bituminous mixture is 110 lb. (figure 9). The test data

TABLE V. MINIATURE BEARING STRENGTH OF SOIL NO. 1* STABILIZED WITH VARIOUS ADDITIVES

Type	Additive Amount (percent of dry soil)	Method of curing	Miniature bearing strength, lb. (after 7 days immersion)
Portland cement, Type I	15	7 days in moist cabinet at approximately 70° F.	2020
Arquad 2HT†	0.5	Same as above	116
Cut-back asphalt (MC-2)	8	No curing of specimen	110

*See table I for properties of the soil.
†A product of Armour and Company, Chicago, Illinois.

of soil No. 1 stabilized with several kinds of additives are given in table V.

The procedure of the Miniature Bearing Test is not as simple as that of the unconfined compression test. The main advantage of the test is that it can be used to evaluate the stability of raw or stabilized soils which have very low or no compressive strength when they are tested by the unconfined compression test after complete immersion in water. The Miniature Bearing Test was found very useful in evaluating the effectiveness of such waterproofing agents as bituminous materials for the stabilization of silty and clayey soils.

SUMMARY

1. The four test methods presented were developed primarily for exploratory or preliminary evaluations of the effectiveness of various additives for the stabilization of silty and clayey soils. The use of such tests makes it possible to screen a large number of additives economically and to select promising ones for further investigations.

2. The density test using 2 in. diameter by 2 in. high specimens gives results fairly close to those obtainable by the standard Proctor density test. It requires only about one-tenth of the material and one-third of the time needed for performing the standard Proctor density test.

3. The flexural test is simple in procedure and requires only a small quantity of material. It is suitable especially for exploratory study purposes.
4. The unconfined compression test may be used for the preliminary evaluation of stabilized soils. The use of 2 in. diameter by 2 in. high specimens is mainly for convenience in preparing the specimens.
5. The Miniature Bearing Test may be used to evaluate the strength of stabilized soils in a manner similar to that of the California bearing ratio test. The Miniature Bearing Test requires only about one-twentieth of the material and one-fourth of the time needed for performing the California bearing ratio test.

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RELATIONSHIP BETWEEN THE CALIFORNIA BEARING RATIO AND THE IOWA BEARING VALUE

by

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(Progress Report, 1956)

INTRODUCTION

One of the difficulties in soil stabilization studies is the inadequacy of testing procedures for determining an exact performance rating of a soil — or an improved soil — for highway pavement design purposes. A commonly used procedure, which attempts to determine this rating by testing the resistance of a soil sample to penetration, is the California Bearing Ratio (CBR) test. Though the CBR test for pavement design purposes seems to be quite reliable, the test procedure is relatively difficult and time-consuming. Also, the test requires fairly large quanties of soil and stabilization materials. The Iowa Bearing Value (IBV) test, a miniature penetration resistance test, is being developed to overcome these disadvantages in testing fine-grained soils.

Since the CBR test has been extensively correlated with the field performance of soils, it is desirable to take advantage of this and to determine the relationship between the California Bearing Ratio and the Iowa Bearing Value.

REVIEW OF PREVIOUS RESEARCH

Development of CBR

A method of highway design, based on a penetration test, called the California Bearing Ratio (CBR) test, was adopted in 1938 after extensive correlation between results of the test and field performance of various California soils[13]. The CBR was adopted for two reasons:

No other single test could be so easily correlated with soil performance for road building purposes.

It was considered vital for highway design purposes to know the penetration resistance of the soil being tested, both in the original compaction condition and after saturation with water. It was also important to know what expansion could be expected in a soil during saturation. The CBR test was the only one which gave all of this information.

During World War II, the U. S. Army Corps of Engineers adopted the CBR design procedure for all military construction of flexible pavements, including hardstands, runways, and roads. The Corps of Engineers performed extensive tests, correlating field performance of additional soil types, refining the test procedure, and expanding the design method to include the greater wheel loads and other special problems encountered in airfield design[1].

The CBR test and design method

The CBR test is performed by causing a flat-ended steel piston of 3 square inch circular cross-sectional area to be pushed into the top surface of a prepared soil specimen at a specified rate. Load readings are taken on either a testing machine dial or some other load reading device at penetrations of 0.025, 0.030, 0.075, 0.10, 0.20, 0.30, 0.40, and 0.50 inch. The readings at these penetrations are then divided by readings taken on a standard "ideal" soil. The California Bearing Ratio is the ratio of the load for the soil being tested to the load for the standard soil at the same penetration, this ratio expressed as a percentage. The test can be performed in a laboratory on undisturbed specimens, or in the field on "in-place" soil.

Details of the CBR design procedure for flexible pavement design can be found in most highway engineering texts[8, 12, 13]. In brief, the procedure is to select from empirical curves the minimum thickness of material which should be placed over the subgrade or base course material whose CBR is known. Design thicknesses for each layer are selected, starting at the lowest limit of construction and working upward to the surface. The design curves are supplemented by tables of minimum design specifications. These prohibit the use of soils of low CBR immediately below the surface layer and establish compaction tolerances. During construction this system may be used as a simple standard inspection test. From the results of one CBR testing the inspector has a good rating of the quality of construction, that is, a comparison of the "in-place" penetration resistance of the soil to that upon which the design was based.

Acceptance of the CBR

The CBR test is one of the most widely used of the common engineering soil tests, and the CBR design method is probably the one most commonly used in flexible pavement design. In 1953, 19 of the 48 states used the CBR in soils testing and classification for design of flexible pavements. Of the 27 states which regularly make soil strength tests, 16 used the CBR as a strength standard[10].

Variations in the CBR test method

To this date, there is no universally accepted procedure for performance

of the CBR test. Each author gives a slightly different approach and testing technique. Several of the more important differences are as follows[2,4,6,12,13,16,17,19]:

1) There is some question concerning what the exact purpose of the test is, and how this purpose affects the testing procedure. For example, some users say that the soaked test is far more important than the unsoaked. They reason that the design should be based on the presumption that the soil will eventually reach a state approaching that of complete saturation. Others say that the soaked test is too severe, since under a pavement soils seldom approach complete saturation.
2) Most use the test load at either 0.10 or 0.20 inch penetration for computing the California Bearing Ratio. However, there is some disagreement as to which of these two penetrations should be used.
3) There is disagreement as to the best apparatus for performing the test.

The American Society for Testing Materials (ASTM) is now engaged in a project which, it is hoped, will result in the adoption of a standard test procedure. The ASTM manual *Procedures for Testing Soils* (1950) presents 2 alternate procedures[16,17]. The first of these two methods, with exceptions listed under Laboratory Investigation below, is the one used in this study.

Need for a simpler test

A research project requiring from 50 to 100 CBR tests would require many man-hours of sample preparation, soaking, and testing times[16,17]. The penetration test itself requires the services of two persons for a minimum of ten minutes actual penetration time. The equipment is heavy, clumsy, space-consuming, and relatively expensive, considering the large number of complete sets required in a well-equipped laboratory. But, since the CBR is a widely accepted standard, any detailed study of the highway and airfield construction characteristics of a soil should include many of the tests. In an evaluation study of soil stabilization, for example, to rate adequately the bearing characteristics of the soils after various curing and simulated weathering treatments, over 100 CBR tests might be required. This many CBR tests is usually out of the question; for this reason, bearing capacity tests are often either inadequately covered or excluded altogether from stabilization studies. Such tests as the unconfined compression test give useful relative strength values but have no established relation to the CBR or to field performance.

Development of IBV

To overcome these disadvantages, the Iowa Bearing Value test is being developed at the Iowa Engineering Experiment Station. This test is to be used with fine-grained soils. Because of the small size of the specimen used, the test will probably not be usable with soils containing material coarser than the No. 10 sieve. However, since most stabilization work is concerned with improving fine-grained soils, this test should be valuable. The recom-

mended procedure for performance of the IBV test is presented in the Appendix.

The relationship between the CBR and IBV has been determined and the CBR of natural or stabilized soils can be predicted from the results of the IBV test. In addition to its potential use in soil stabilization research and design, the IBV test may also be useful as a construction control test. For example, inspection can be more thorough and less time-consuming if the IBV is used instead of the CBR in routine checks on the quality of construction.

Various other tests have been correlated with the CBR. For example, the Mississippi River Commission's Waterways Experiment Station has correlated its own Corps of Engineers Cone Index with the CBR, and has made extensive studies on the correlation between CBR and moisture content and density[7,18].

LABORATORY INVESTIGATION

General Procedure

In the laboratory investigation for correlating results of the CBR and IBV tests, the correlation tests had two objectives:

1) CBR load readings are used chiefly at either 0.10 or 0.20 inch penetration in computing the CBR. Therefore, any system for predicting the CBR from another test, such as the IBV test, should provide for predicting both the unsoaked and soaked CBR at both of these penetrations. Results of the correlation tests must make it possible to decide which penetrations of the IBV test correlate best with those used in defining the CBR.

2) Since both the CBR and IBV specimens are at about the same density, and therefore have about the same permeability, it is to be expected that the IBV specimen, being smaller, will take less time to reach any given degree of saturation than the larger (CBR) specimen. A suitable soaking time for the IBV soaking test must be chosen. The soaking time of 96 hours is the most commonly used in the CBR test. Therefore, results of the correlation tests must make it possible to decide which IBV soaking time (presumably less than 96 hours) yields results which best correlate with those of the 96-hour soaked CBR test.

Considering these goals, the following general investigation procedure was planned:

1) *Unsoaked tests.* For each soil, mix, at optimum moisture content, enough material for 2 CBR specimens and 3 IBV specimens. Mold these specimens. Make density and moisture content determinations on each specimen. Test each of these specimens as indicated in the CBR and IBV test methods presented below. Determine the CBR at 0.10 and 0.20 inch penetrations, and the IBV test load at 0.02, 0.04, 0.06, 0.08, 0.10, 0.12, 0.14, 0.16, 0.18, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, and 0.50 inch penetrations.

2) *Soaked tests.* For each soil, mix, at optimum moisture content, enough material for 2 CBR specimens and 9 IBV specimens. Mold these specimens. Make density and moisture content determinations on each specimen at time of molding. Soak all CBR specimens for 96 hours; and soak three IBV specimens each, for 24, 48, and 72 hours. Make a determination of the amount of moisture absorbed by each specimen after completion of its soaking period. Take expansion readings on all soaking specimens immediately after immersion, and again at the end of each 24 hours during the soaking period. Test each specimen after completion of its soaking period. Record CBR's and IBV test loads at the same penetrations used for the unsoaked tests, above.

TABLE I. SAMPLING LOCATION OF SOILS

Sample No.	Iowa County	Township	Range	Section	Reference
20-2-III and 20-2-IV*	Harrison	T-78N	R-44W	S-15	9
100-8	Scott	T-77N	R-2E	S-13	9
26-1	Shelby	T-81N	R-40W	S-21	9
44-A-1	Page	T-68N	R-39W	S-21	None
Luton clay	Missouri River Floodplain†				14
411	Page	T-69N	R-36W	S-27	None
79-S	Mahaska	T-74N	R-16W	S-25	21
CS	Story	T-83N	R-24W	S-1	None
S-26-1	Blended of CS and 26-1, in the ratio 9:7 by weight				
FS-26-1	Blended of 79-S and 26-1, in the ratio 2:1 by weight				

*Samples 20-2-III and 20-2-IV were sampled in the same location, 20-2-IV several months after 20-2-III.
†No other information available on this location.

TABLE II. PROPERTIES OF SOIL SAMPLES

	SAMPLE NUMBERS										
Property	S-26-1	20-2-III	100-8	26-1	FS-26-1	44-A-1	Luton clay	20-2-IV	411	79-S	CS
L.L., %	29.5	33.2	27.1	39.4	17.2	53.1	71.0	32.2	41.8	non-plastic	
P.L., %	19.2	25.6	19.8	26.9	13.2	25.7	24.5	23.0	14.9	....	....
P.I., %	10.3	7.6	7.3	12.5	4.0	27.4	46.5	9.2	26.9	....	....
C.M.E., %	13.7	13.1	8.7	19.5	7.1	15.2	38.4	19.6	21.7	1.4	2.0
F.M.E., %	25.6	30.7	27.6	32.6	26.4	50.6	48.7	26.0	31.8	23.1	20.5
S.L., %	19.7	26.2	20.6	23.3	14.3	19.9	10.2	24.5	12.3	12.6	0.0
C.E.C. me/100 g.	7.3	12.6		15.3	6.8	28.2	39.4	13.4	20.0	1.5	13.4
Std. Proctor den., pcf	127.8	108.4	110.6	107.0	127.0	104.0	90.8	108.4	115.2	109.3	113.3
O.M.C., %	10.4	18.0	15.8	17.7	9.8	19.5	23.0	18.0	15.5	12.7	15.6
Mech. Anal.*											
sand, %	60.5	0.3	2.8	2.0	63.0	0.2	1.5	0.4	32.7	95.6	92.5
silt, %	28.0	82.2	85.2	70.6	26.1	58.0	24.2	81.2	30.8	0.7	4.2
clay, %	11.5	17.5	12.0	27.4	10.9	41.8	74.3	18.4	36.5	3.7	3.3
coll., %	8.7	15.0	8.9	20.0	8.7	31.0	55.5	15.1	26.0	2.6	2.9
BPR Class. Engr. Textural	A-4(1) sandy loam	A-4(8) silty loam	A-4(8) silty loam	A-6(9) silty clay	A-4(0) sandy loam	A-7-6(17) silty clay	A-7-6(20) clay	A-4(8) silty loam	A-7-6(13) clay	A-3(0) sand	A-3(0) sand

*Sand — 2.0 to 0.074 mm; silt — 0.074 to 0.005 mm; clay — smaller than 0.005 mm; colloidal clay — smaller than 0.001 mm.

Soil Samples

Samples of nine Iowa soils were used in this study. These were chosen to represent sandy, silty, and clayey soils (tables I, II). Two of the samples, S-26-1 and FS-26-1, are not naturally occurring soils; each is a blend of two natural soils. S-26-1 is a mixture of a coarse sand (CS) and a medium-textured loess (26-1) in the ratio 9:7 by weight; FS-26-1 is composed of a fine sand (79-S) and the same loess in the ratio 2:1 by weight. These blends were used because the sands alone did not have enough cohesion to give consistent test results.

Method of Testing

CBR tests. The CBR test procedure used is one of the two procedures recommended in ASTM Manual[17, p. 386], with the following exceptions:

All specimens were compacted using standard Proctor compactive effort: 35 blows of a 10 pound hammer, with an 18 inch drop, on each of three layers.

All soaked CBR test samples were soaked 96 hours. A 10 pound surcharge rested on each specimen during the soaking period.

Surcharges were removed after soaking, before the penetration test was performed. The reason for this was as follows: The surcharge during testing is to simulate the confining effect of a surface layer. However, it is felt that confinement of the soil by the walls of the mold cylinder is at least as great as that which would result, in the field, due to the total confinement of surfacing and adjacent soil. Therefore, no additional surface load on the specimen is desired.

All tests were performed on remolded specimens.

Bearing ratios were computed at both 0.10 and 0.20 inch penetrations. This was to allow comparison of IBV test results, since authorities disagree as to the exact penetration by which the CBR should be defined.

IBV tests. Except as indicated, the IBV test procedure used throughout this study was as given in the Appendix.

Presentation and Discussion of Test Results

Results of IBV and CBR tests. Tables III and IV show results of IBV and CBR tests obtained as indicated above. In a typical plot of IBV and CBR load-versus-penetration curves the curves representing the three soaked IBV tests are very close together, and the 72 hour curve is above the 48 hour curve (figure 2). Such an arrangement of the curves was not uncommon. Strength tended to increase in several rather than to decrease with soaking time.

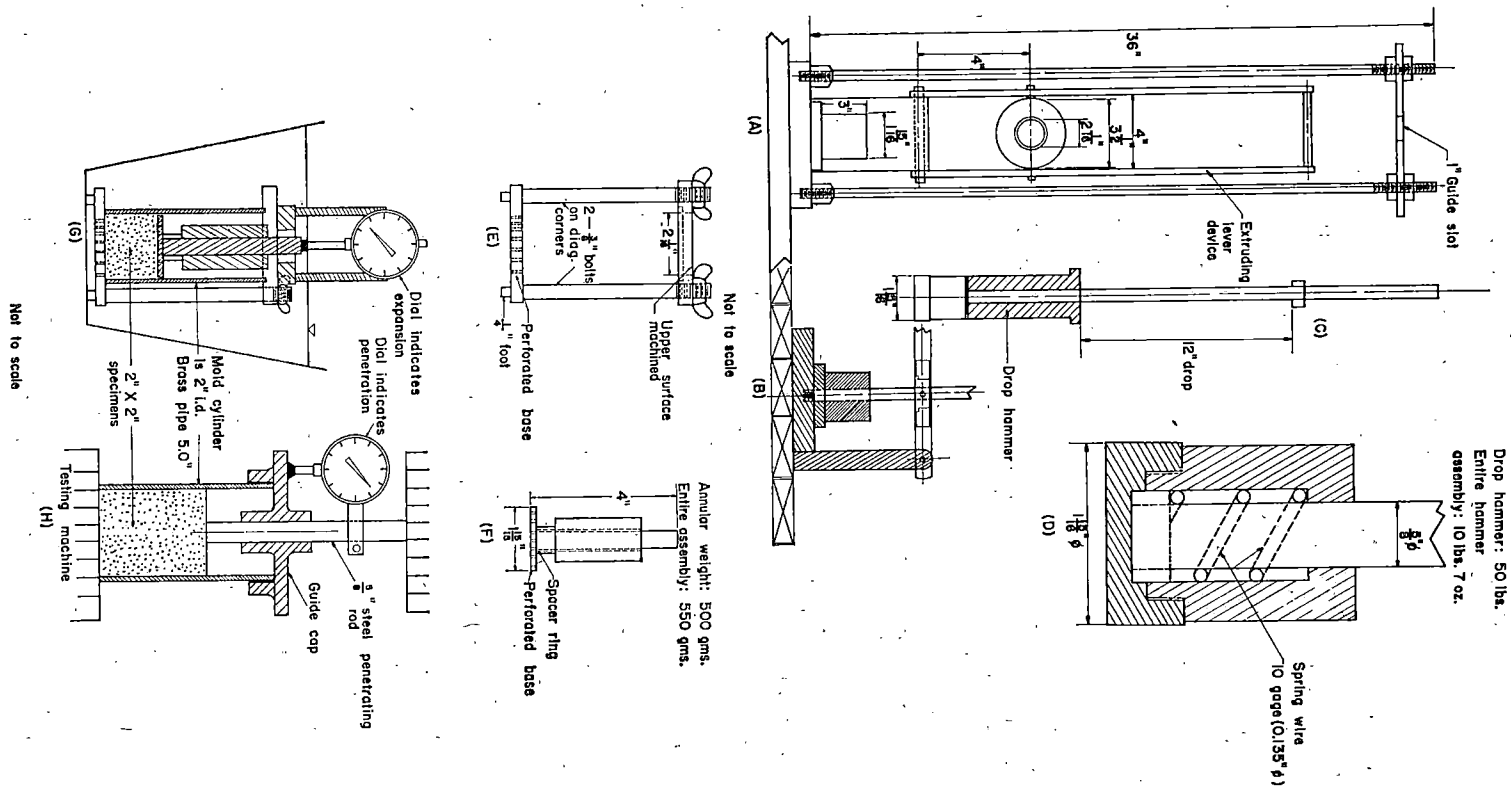

Fig. 1. IBV test apparatus: (A) Molding machine with extruding lever in upright position; (B) Molding machine with extruding lever in horizontal, working position; (C) Drop hammer; (D) Detail of head of drop hammer; (E) Soaking frame; (F) Annular weight assembly; (G) Specimen during soaking test with expansion dial in place; (H) Specimen during penetration test.

TABLE III. RESULTS OF IOWA BEARING VALUE TESTS

Penetration and soaking (in.)	time (hrs.)	SAMPLE NUMBERS S-26-1	20-2-III	100-8	26-1	FS-26-1		44-A-1	Luton clay	20-2-IV	411
						IBV loads, lbs.*					
0.06	00	23	90	57	59	143	124	45	42	88	71
		27	96	57	60	120	125	46	55	90	68
			103	71	58	128	120	52	48	82	70
						Average IBV loads, lbs.					
		25	96.3	61.7	59	126.7		47.7	48.3	86.7	69.7
						Average logarithms					
		1.3966	1.9831	1.7877	1.7708	2.1019		1.6773	1.6816	1.9375	1.8430
						IBV loads, lbs.					
0.06	24	34	57	24	37	87	109	34	7	42	14
		30		20	36	83	98	37	7	41	11
					36	83					13
						Average IBV loads, lbs.					
		32	57	22	36.3	92		35.5	7	41.5	12.7
						Average logarithms					
		1.5043	1.7559	1.3406	1.5603	1.9613		1.5498	0.8451	1.6180	1.1005
						IBV loads, lbs.					
0.06	48	35	53	26	37	100	99	30	5	35	11
		35	66	21	30	104	107	38	2	47	8
					29	99		28			14
						Average IBV loads, lbs.					
		35	59.5	23.5	32	101.8		32	3.5	41	11
						Average logarithms					
		1.5441	1.7719	1.3686	1.5026	2.0075		1.5014	0.5000	1.6081	1.0302
						IBV loads, lbs.					
0.06	72	25	55	7	43	105	106	37	3	48	11
		29	65	21	41	105	87	32	3	48	8
					40		100	33			8
						Average IBV loads, lbs.					
		28	60	14	41.3	100.6		34	3	48	9
						Average logarithms					
		1.4458	1.7766	1.0836	1.6161	2.0014		1.5306	0.4771	1.6812	0.9492
						IBV loads, lbs.					
0.08	00	33	105	72	68	170	152	50	48	105	77
		34	112	73	67	142	150	50	54	108	72
			120	86	65	150	148	57	54	100	77
						Average IBV loads, lbs.					
		33.5	112	77	66.7	152		52.3	54.3	104.3	75.3
						Average logarithms					
		1.5250	2.0499	1.8850	1.8238	2.1812		1.7180	1.7330	2.0182	1.8768
						IBV loads, lbs.					
0.08	24	44	66	28	42	103	100	37	7	51	15
		40		23	42	95	128	40	7	49	13
					42		118				14
						Average IBV loads, lbs.					
		42	66	25.5	42	108.8		38.5	7	50	14
						Average logarithms					
		1.6228	1.8195	1.4044	1.6232	2.0339		1.5852	0.8451	1.6989	1.1454
						IBV loads, lbs.					
0.08	48	45	63	30	44	117	119	33	5	43	13
		45	75	25	35	127	117	40	2	54	10
					34		125	30			17
						Average IBV loads, lbs.					
		45	69	27.5	37.7	121		34.3	3.5	48.5	13.3
						Average logarithms					
		1.6532	1.8372	1.4375	1.5730	2.0825		1.5326	0.5000	1.6830	1.1148
						IBV loads, lbs.					
0.08	72	33	65	11	49	125	116	38	3	57	11
		41	75	25	45	127	126	35	4	57	10
		39			43		102	35			10
						Average IBV loads, lbs.					
		37.7	70	18	45.7	119.2		36	3.5	57	10.3
						Average logarithms					
		1.5741	1.8440	1.2196	1.6590	2.0748		1.5560	0.5396	1.7559	1.0138
						IBV loads, lbs.					
0.10	00	43	119	82	73	197	178	55	58	120	81
		44	127	90	72	163	175	54	67	120	77

*On the average, three tests were performed on each soil; however, as explained in the test, sometimes fewer tests were performed due to limitations in availability of equipment. For soil FS-26-1, data from two sets of tests are included.

TABLE III. (CONTINUED).

Penetration and soaking time (in.)	(hrs.)	SAMPLE NUMBERS									
		S-26-1	20-2-III	100-8	26-1	FS-26-1		44-A-1	Luton clay	20-2-IV	411
			133	102	70	175	173	60	60	114	80
					Average IBV loads, lbs.						
		43.5	128	91.3	71.7	176.8		56.3	61.7	118	79.3
					Average logarithms						
		1.6385	2.1064	1.9589	1.8552	1.2468		1.7503	1.7892	2.0718	1.8994
					IBV loads, lbs.						
0.10	24	55	75	32	47	118	117	38	8	60	15
		51		27	49	109	146	41	8	55	16
					50		137				18
					Average IBV loads, lbs.						
		53	75	29.5	48.7	125.4		39.5	8	57.5	16.3
					Average logarithms						
		1.7240	1.8751	1.4682	1.6871	2.0957		1.5963	0.9031	1.7593	1.2118
					IBV loads, lbs.						
0.10	48	55	71	36	51	135	135	35	5	55	14
		55	87	27	40	148	135	40	2	61	13
					39		140	33			20
					Average IBV loads, lbs.						
		55	79	31.5	43.3	138.6		36	3.5	58	15.7
					Average logarithms						
		1.7404	1.8954	1.4938	1.6336	2.1415		1.5549	0.5000	1.7628	1.1870
					IBV loads, lbs.						
0.10	72	42	74	16	54	143	130	40	4	66	12
		53	86	33	50	149	145	40	5	64	12
		50			46		119	35			11
					Average IBV loads, lbs.						
		48.3	80	24.5	50	137.2		38.3	4.5	65	11.7
					Average logarithms						
		1.6822	1.9018	1.3613	1.6981	2.1359		1.5828	0.6506	1.8128	1.0666
					IBV loads, lbs.						
0.12	00	53	135	94	80	223	205	58	63	134	85
		54	140	101	77	183	195	57	72	130	80
			155	115	76	196	197	62	65	130	85
					Average IBV loads, lbs.						
		53.5	143.3	103.3	77.7	199.8		59	66.7	131.3	83.3
					Average logarithms						
		1.7284	2.1556	2.0133	1.8901	2.2999		1.7706	1.8232	2.1183	1.9206
					IBV loads, lbs.						
0.12	24	66	85	35	53	135	135	40	8	69	15
		60		34	54	121	163	43	8	63	18
					55		152				18
					Average IBV loads, lbs.						
		63	85	34.5	54	141.2		41.5	8	66	17
					Average logarithms						
		1.7988	1.9294	1.5378	1.7324	2.1475		1.6178	0.9031	1.8190	1.2289
					IBV loads, lbs.						
0.12	48	65	80	42	57	152	151	37	5	67	15
		63	97	32	45	166	153	42	2	67	15
					43		158	35			20
					Average IBV loads, lbs.						
		64	88.5	37	48.3	156		38	3.5	67	16.7
					Average logarithms						
		1.8061	1.9450	1.5642	1.6809	2.1929		1.5785	0.5000	1.8261	1.2177
					IBV loads, lbs.						
0.12	72	49	85	22	55	164	145	42	4	74	12
		64	97	42	55	168	164	43	5	70	14
		69			51		135	38			12
					Average IBV loads, lbs.						
		60.7	91	32	53.7	155.2		41	4.5	72	12.7
					Average logarithms						
		1.7784	1.9581	1.4828	1.7295	2.1893		1.6122	0.6506	1.8572	1.1015
					IBV loads, lbs.						
0.14	00	62	148	106	85	247	225	60	68	146	88
		63	155	116	83	201	220	60	75	145	83
			169	128	82	218	222	66	69	145	87
					Average IBV loads, lbs.						
		62.5	157.3	116.7	83.3	222.2		62	70.7	145.3	86
					Average logarithms						
		1.7958	2.1962	2.0657	1.9208	2.3459		1.7920	1.8488	2.1624	1.9344
					IBV loads, lbs.						
0.14	24	77	94	39	57	150	153	43	8	79	16
		66		39	60	136	179	45	8	70	20
					64		171				18

TABLE III. (CONTINUED).

Penetration (in.)	Soaking time (hrs.)	SAMPLE NUMBERS								
		S-26-1	20-2-III	100-S	26-1	FS-26-1	44-A-1	Luton clay	20-2-IV	411
		Average IBV loads, lbs.								
		71.5	94	39	60.3	157.8	44	8	74.5	18
		Average logarithms								
		1.8530	1.9731	1.5911	1.7801	2.1960	1.6434	0.9031	1.8714	1.2535
		IBV loads, lbs.								
0.14	48	72	90	46	63	169 169	40	6	70	15
		70	106	37	49	183 171	44	2	74	15
					47	175	37			20
		Average IBV loads. lbs.								
		71	98	41.5	53	173.4	40.3	4	72	16.7
		Average logarithms								
		1.8512	1.9898	1.6155	1.7205	2.2389	1.6046	0.5396	1.8572	1.2177
		IBV loads, lbs.								
0.14	72	56	93	29	58	183 161	45	4	83	13
		73	108	46	57	185 180	45	5	78	14
		63			59	150	40			13
		Average IBV loads, lbs.								
		65.7	100.5	37.5	58	171.8	43.3	4.5	80.5	13.3
		Average logarithms								
		1.8147	2.0010	1.5626	1.7634	2.2336	1.6362	0.6506	1.9056	1.1246
		IBV loads, lbs.								
0.16	00	68	160	118	92	270 247	64	74	160	90
		69	168	130	87	223 241	61	77	160	85
			183	144	88	240 242	69	74	160	90
		Average IBV loads, lbs.								
		68.5	170.3	130.7	89	243.8	64.7	75	160	88.3
		Average logarithms								
		1.8356	2.2306	2.1147	1.9493	2.3864	1.8101	1.8750	2.2041	1.9459
		IBV loads, lbs.								
0.16	24	65	104	45	63	164 171	45	8	88	17
		73		46	63	148 196	46	8	79	23
					69	189				18
		Average IBV loads, lbs.								
		79	104	45.5	65	173.6	45.5	8	83.5	19.3
		Average logarithms								
		1.8964	2.0170	1.6580	1.8125	2.2374	1.6580	0.9031	1.9210	1.2825
		IBV loads, lbs.								
0.16	48	80	100	55	67	186 188	43	6	77	15
		77	115	43	53	201 188	46	3	84	15
					50	190	39			20
		Average IBV loads, lbs.								
		78.5	107.5	49	56.7	190.6	42.7	4.5	80.5	16.7
		Average logarithms								
		1.8948	2.0304	1.6870	1.7498	2.2800	1.6291	0.6276	1.9054	1.2177
		IBV loads, lbs.								
0.16	72	64	101	35	66	198 175	46	5	91	14
		81	117	55	64	200 196	45	5	85	14
		76			65	164	42			14
		Average IBV loads, lbs.								
		73.7	109	45	65	186.6	44.3	5	88	14
		Average logarithms								
		1.8652	2.0362	1.6422	1.8129	2.2696	1.6464	0.6990	1.9442	1.1461
		IBV loads, lbs.								
0.18	00	76	172	129	97	294 269	66	78	175	92
		76	182	144	90	242 265	65	80	175	87
			193	159	94	263 266	72	78	173	93
		Average IBV loads, lbs.								
		76	184	144	93.7	266.5	67.7	78.7	174.3	90.7
		Average logarithms								
		1.8808	2.2641	2.1568	1.9714	2.4250	1.8299	1.8958	2.2413	1.9573
		IBV loads, lbs.								
0.18	24	92	113	52	67	175 189	48	8	97	19
		78		54	66	161 210	49	8	87	25
					75	204				19
		Average IBV loads, lbs.								
		85	113	53	69.3	187.8	48.5	8	92	21
		Average logarithms								
		1.9280	2.0531	1.7242	1.8402	2.2716	1.6857	0.9031	1.9632	1.3185
		IBV loads, lbs.								
0.18	48	86	109	55	70	205 208	45	6	87	16

TABLE III. (CONTINUED).

Penetration and soaking time (in.)	(hrs.)	SAMPLE NUMBERS S-26-1	20-2-III	100-S	26-1	FS-26-1		44-A-1	Luton clay	20-2-IV	411
		85	124	48	57	218	204	49	3	94	15
		Average IBV loads, lbs.									
		85.5	116.5	51.5	61	203.8		44.7	4.5	90.5	17
		Average logarithms									
		1.9320	2.0654	1.7108	1.7831	2.3196		1.6485	0.6276	1.9563	1.2271
		IBV loads, lbs.									
0.18	72	70	109	42	70	214	189	46	7	101	15
		88	127	65	67	214	210	46	7	91	14
		84			69		180	47			15
		Average IBV loads, lbs.									
		80.7	118	53.5	68.7	201.4		46.3	7	96	14.7
		Average logarithms									
		1.9046	2.0706	1.7180	1.8367	2.3030		1.6659	0.8451	1.9816	1.1661
		IBV loads, lbs.									
0.02†	00	7	51	24	33	70	55	30	28	47	50
		8	53	12	31	63	53	30	39	48	48
			54	33	33	63	50	37	34	40	50
		IBV loads, lbs.									
0.02	24	12	31	13	19	40	35	26	4	18	6
		5		8	19	46	55	25	5	19	7
					16		45				5
		IBV loads, lbs.									
0.02	48	12	28	13	21	47	52	22	5	15	5
		12	36	11	15	49	50	25	1	14	4
					15		58	20			8
		IBV loads, lbs.									
0.02	72	7	24	6	24	28	50	25	1	26	10
		6	30	10	27	55	52	20	2	20	5
		7			24		45	27			7
		IBV loads, lbs.									
0.04	00	14	72	44	47	114	97	39	36	70	64
		17	73	40	48	95	95	40	49	73	60
			83	56	48	99	88	47	41	63	63
		IBV loads, lbs.									
0.04	24	20	46	20	30	72	63	31	7	32	10
		16		16	30	67	87	35	6	32	10
					29		75				10
		IBV loads, lbs.									
0.04	48	23	42	21	33	78	78	26	5	26	8
		24	52	17	26	84	77	33	1	33	6
					24		89	25			12
		IBV loads, lbs.									
0.04	72	15	34	7	37	84	82	35	2	40	11
		15	52	16	37	83	84	27	3	38	6
		15			35		68	32			8
		IBV loads, lbs.									
0.20	00	84	186	142	102	316	289	68	83	188	93
		86	195	158	95	261	286	67	84	185	89
			213	173	93	280	287	74	84	190	95
		IBV loads, lbs.									
0.20	24	100		58	74	187	208	50	8	106	21
		86		58	70	169	225	51	8	95	25
		93	122		80	222					20
		IBV loads, lbs.									
0.20	48	99	118	59	73	226	228	49	6	98	17
		92	124	52	61	234	222	50	4	102	16
					63		224	43			20
		IBV loads, lbs.									
0.20	72	79	117	46	73	225	202	49	8	108	18
		93	137	70	71	229	227	49	8	101	14
		91			70		196	50			18

†Loads are given only for the penetrations 0.02, 0.04, 0.20, 0.30, 0.40, and 0.50 inch. These figures were recorded during testing, but were not used in correlating results of the CBR and IBV tests. Loads at 0.25, 0.35, and 0.45 inch penetration were recorded, but they are not needed to draw smooth penetration-versus-load curves, and are not included.

TABLE III. (CONTINUED).

Penetration and soaking (in.)	time (hrs.)	SAMPLE NUMBERS S-26-1	20-2-III	100-8	26-1	FS-26-1		44-A-1	Luton clay	20-2-IV	411
						IBV loads, lbs.					
0.30	00	125	248	194	120	407	387	83	94	250	103
		130	259	222	114	348	386	76	100	250	101
			278	236	116	375	382	83	93	256	105
						IBV loads, lbs.					
0.30	24	140	162	99	93	244	277	65	9	150	33
		120		99	90	220	295	65	8	136	31
					100		294				35
						IBV loads, lbs.					
0.30	48	121	162	87	90	309	300	60	7	143	25
		126	182	82	85	306	290	59	5	152	24
					80		290	53			30
						IBV loads, lbs.					
0.30	72	106	159	79	94	289	257	63	10	153	25
		123	189	106	95	297	285	57	10	145	20
		121			93		260	59			24
						IBV loads, lbs.					
0.40	00	145	309	236	140	470	457	99	108	297	113
		152	320	254	136	423	456	85	107	300	110
			345	240	134	450	463	89	106	310	112
						IBV loads, lbs.					
0.40	24	173	200	144	110	280	333	70	12	178	43
		149		154	110	260	351	72	10	178	43
					122		329		11		37
						IBV loads, lbs.					
0.40	48	148	202	120	117	373	338	67	9	185	32
		152	227	120	100	337	325	67	6	210	30
					95		342	63			35
						IBV loads, lbs.					
0.40	72	132	196	114	115	331	288	65	11	206	29
		155	232	147	112	351	324	70	10	182	30
		151			115		292	68			27
						IBV loads, lbs.					
0.50	00	168	368	237	155	528	518	100	112	355	123
		182	390	263	146	487	526	89	118	370	116
			416	240	152	513	532	96	115	396	122
						IBV loads, lbs.					
0.50	24	205	227	187	130	309	374	75	12	209	53
		185		188	122	289	382	79	11	218	51
					142		366				51
						IBV loads, lbs.					
0.50	48	171	238	147	127	415	370	75	12	234	39
		179	263	152	112	383	352	73	8	254	38
					112		376	75			45
						IBV loads, lbs.					
0.50	72	158	228	138	125	370	317	75	15	256	40
		181	278	186	127	394	350	74	15	214	34
		174			127		323	76			36

TABLE IV. RESULTS OF CALIFORNIA BEARING RATIO TEST

Penetration and soaking time (in.)	(hrs.)	SAMPLE NUMBERS S-26-1	20-2-III	100-8	26-1	FS-26-1	44-A-1	Luton clay	20-2-IV	411
					CBR values, %*					
0.10	00	4.20	15.37	8.47	10.83	15.30	11.53	12.67	8.49	15.73
		4.00	14.17	8.20	10.50	15.60	10.67	9.80	99.01	15.00
					Average CBR, %					
		4.10	14.77	8.37	10.66	15.45	11.10	11.23	8.75	15.36
					Average logarithms					
		0.6126	1.1690	0.9208	1.0279	1.1888	1.0450	1.0470	0.9418	1.1864
					CBR values, %					
0.10	96	4.70	9.80	6.23	7.23	20.60	5.33	2.13	11.93	2.67
		4.27	9.60	5.63	5.30	20.60	4.67	1.53	7.20	2.63
					Average CBR, %					
		4.48	9.70	5.93	6.26	20.60	5.00	1.83	9.56	2.65
					Average logarithms					
		0.6512	0.9862	0.7725	0.7917	1.3139	0.6980	0.2566	0.9670	0.4232
					CBR values, %					
0.20	00	5.49	20.55	11.35	10.90	20.60	10.00	12.00	11.50	13.10
		5.09	18.75	11.00	10.83	20.50	9.22	9.38	11.30	12.48
					Average CBR, %					
		5.29	19.65	11.18	10.86	20.55	9.61	10.69	11.40	12.79
					Average logarithms					
		0.7232	1.2929	1.0482	1.0360	1.3128	0.9824	1.0257	1.0569	1.1068
					CBR values, %					
0.20	96	5.94	11.00	8.26	7.06	27.25	4.67	1.55	12.70	2.62
		5.65	11.50	7.53	5.21	27.00	4.00	1.49	9.37	2.34
					Average CBR, %					
		5.79	11.25	7.89	6.14	27.12	4.34	1.52	11.01	2.48
					Average logarithms					
		0.7625	1.0510	0.8968	0.7828	1.4334	0.6357	0.1818	1.0378	0.3940

*In all cases, 2 CBR tests were performed on each soil.

TABLE V. COMPARISON OF COMPACTED DRY DENSITIES OF CBR AND IBV SPECIMENS

Sample No.	Specimens used in unsoaked tests CBR	IBV	Specimens used in soaked tests CBR	IBV
S-26-1	126.0 pcf	127.3 pcf	124.6 pcf	128.8 pcf
20-2-III	105.0	104.1	104.6	105.4
100-S	10.7	108.0	107.6	110.3
26-1	105.6	109.3	106.6	107.6
FS-26-1	125.2	127.3	124.6	125.6
44-A-1	100.5	104.0	99.9	105.4
Luton Clay	87.9	90.7	89.3	93.0
20-2-IV	105.8	106.6	105.5	106.7
411	109.2	115.5	110.2	116.4

Density data. Table V shows densities of IBV and CBR specimens after compaction. For several of the soils, notably those of high clay content (26-1, 44-A-1, Luton Clay, 411), IBV densities are higher than CBR densities by about 5 percent. This is probably due to the plasticity of these high clay soils. The head of the drop-hammer used in preparing the CBR specimen is much smaller in cross-section than the CBR mold cylinder, but the hammer used in preparing the IBV specimen has practically the same cross-sectional area as the inside of the IBV mold cylinder. Therefore, while the CBR hammer is compacting the specimen, a considerable amount of kneading occurs; this kneading action does not take place in preparing the IBV specimen. As highly plastic soils are compacted in the IBV mold, plastic flow

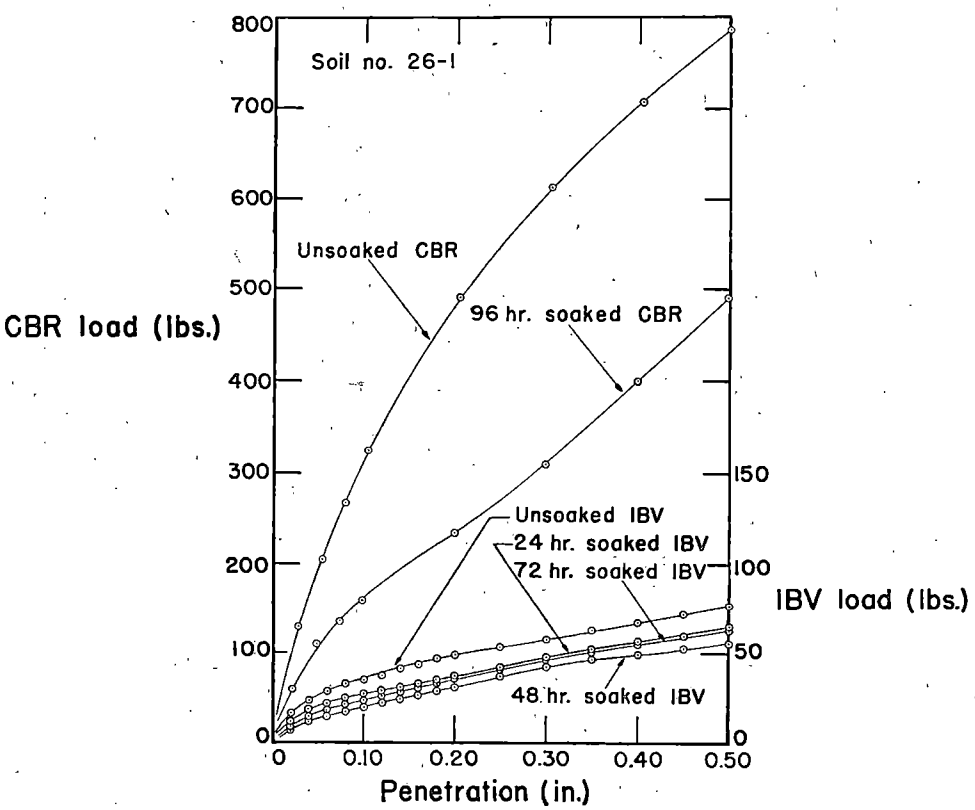

Fig. 2. Typical CBR and IBV load versus penetration curves.

compensates for the lack of kneading. With less plastic soils less plastic flow occurs and compacted density is less affected. The IBV compaction procedure could possibly be changed to allow less compactive effort on soils of high plasticity. This subject will be mentioned again under Suggestions for Future Research.

Remarks concerning required testing penetration. Very often, it is necessary to correct the CBR load-versus-penetration curve for irregularities in the penetration resistance at low penetration (figure 3)[15, 17]. This is done whenever the lower penetration end of the curve is concaved upwards. As can be seen from this figure, the correction requires that the specimen be penetrated deeped than 0.20 inch — even though for most tests, load readings beyond this penetration are not needed. In none of the IBV tests of this study was such a correction needed. Since the correction is not needed, penetration of the sample to depths greater than 0.20 inch is not necessary. The IBV specimen may be penetrated to only 0.20 inch depth, instead of the 0.50 inch depth required for the CBR test. Since the speed of penetration is 0.05 inch per minute, approximately six minutes of testing time will be saved

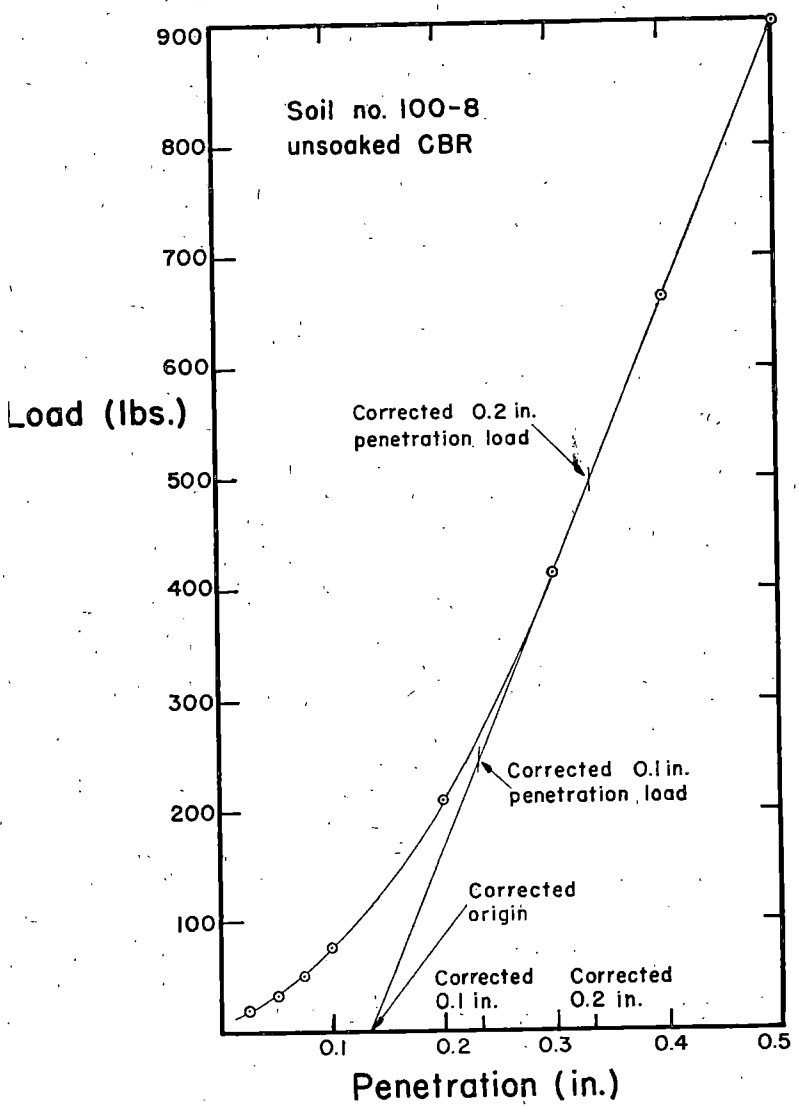

Fig. 3. Correction of CBR load versus penetration curve for surface irregularities. Tangent is drawn from steepest part of the curve.

per test, reducing penetration time from ten to four minutes per IBV test.

Expansion data. Expansion data is given in percent of original volume (volume immediately after molding) for CBR and IBV specimens after 24, 48, 72, and 96 hours soaking (table VI). From this data all but two of the soils are relatively non-expansive. That is, expansion of both CBR and IBV specimens does not exceed 1 percent. The two soils in which considerable expansion occurs are Luton Clay and 411. In general, the percentage expansion

TABLE VI. RESULTS OF IBV AND CBR EXPANSION TESTS

Soaking time (hrs.)	SAMPLE NUMBERS								
	S-26-1	20-2-III	100-8	26-1	FS-26-1	44-A-1	Luton clay	20-2-IV	411
	Expansion of IBV specimens*, %								
24	0.09	0.52	0.61	0.96	0.04	0.65	7.50	0.51	2.70
48	0.10	0.53	0.62	0.98	0.05	0.95	8.10	0.52	3.52
72	....†	0.54	0.65	0.99	0.67	1.05	8.52	0.52	4.10
	Expansion of CBR specimens, %								
24	0.00	0.39	0.13	1.00	0.02	0.54	3.55	0.22	1.35
48	0.00	0.45	0.16	1.06	0.02	0.80	4.01	0.23	1.71
72	0.03	0.45	0.18	1.10	0.02	0.82	4.26	0.24	1.93
96	0.03	0.45	0.19	1.12	0.02	0.86	4.39	0.24	2.13

*All expansion data are expressed in percent of original height of specimen immediately after compaction.
†Not recorded due to an accident in handling.

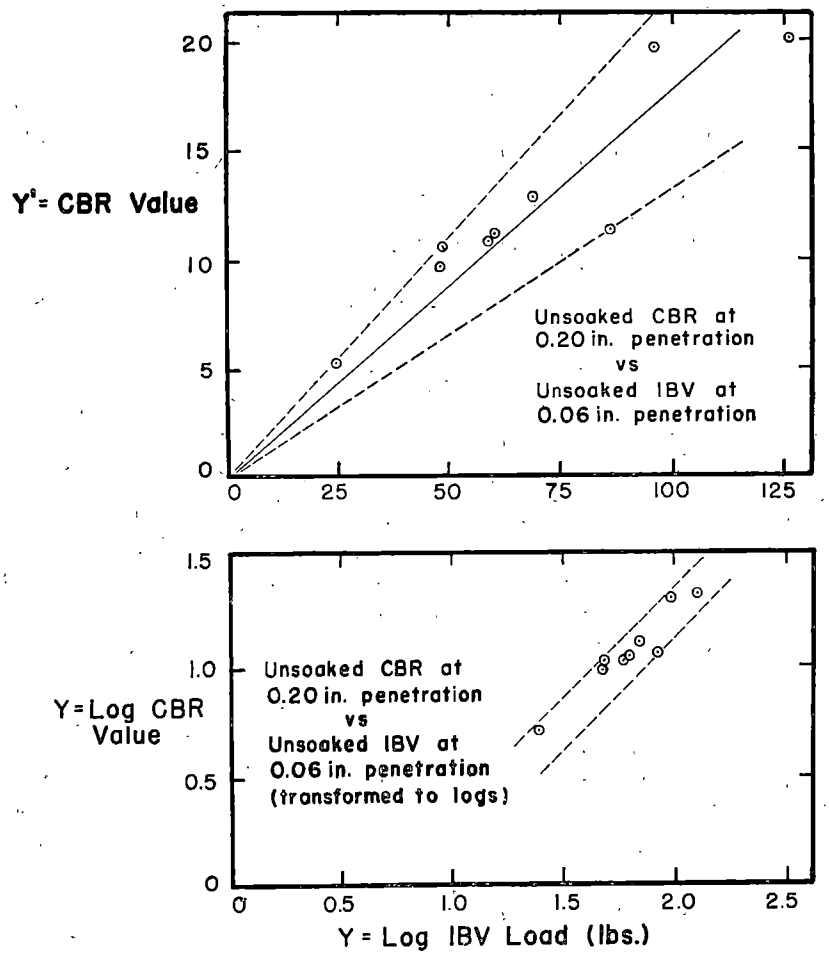

Fig. 4. Transformation to logarithms. Upper graph shows relationship between CBR and IBV test results. Lower graph shows this relationship with results of both tests transformed to logarithms.

of the IBV specimens for these soils is roughly twice that of the CBR specimens. Although this ratio might serve as a rough guide to prediction of CBR expansion from results of IBV expansion tests, an exact correlation of the expansion of the IBV and CBR specimens would require additional data.

STATISTICAL ANALYSIS

Organization of the Analysis

General. On plotting IBV readings at a given penetration versus CBR values for either 0.10 or 0.20 inch penetration, it is apparent that there is some relationship between the results of the two tests (figure 4). On this figure, a straight line seems to represent the approximate linear relationship. However, to decide which combinations of IBV and CBR data are the best for prediction, and to know the error to be expected in a prediction of the CBR from a given number of IBV tests, a detailed statistical analysis is required.

In this statistical analysis it is assumed that a true *straight line* relationship exists. This line can be represented by the equation

$$Y' = \alpha' X' + \beta'$$

where Y' is the true CBR value for a given soil, X' is the true IBV value for the soil, α' is the slope of the line, and β' is the Y' intercept of the line. However, due to the inexactness (lack of reproducibility) of both tests, it is impossible to measure the true X' or true Y' values for the soil without making a very large number of tests — theoretically, an infinite number. Therefore, the observed measurement, x' or y', obtained from any single test, is the true value plus an error. Thus

$$x' = X' + e' \text{ and } y' = Y' + f'$$

Transformation to logarithms. Before applying appropriate statistical techniques, a transformation of data is necessary. The only statistical procedures available assume a uniform spread of errors along the line, but the spread is not uniform (figure 4). It is much greater in the upper regions of the graph than in the lower. However, the spread does seem to be directly proportional to the x' and y' values, the points forming a cone with apex at the origin. If it is assumed:

(1) that the errors in x' and y' are directly proportional to the magnitude of X' and Y', and

(2) that the line passes through the origin ($\beta = 0$),

then $x' = X + e'$ becomes $x' = X + aX'$,

and $y' = Y' + f'$ becomes $y' = Y' + bY'$;

where a and b are random quantities which have expected value equal to zero. The equation for the line becomes approximately

$$Y'(1 + b) = \alpha' X'(1 + a)$$

Taking logarithms of both sides yields:

$$\log Y' + \log (1+b) = \log X' + \log (1+a) + \log \alpha'$$

Now define: $Y = \log Y'$ $\quad e = \log (1+a)$

$X = \log X'$ $\quad f = \log (1+b)$

Here, e and f are random quantities, which, it is reasonable to assume, have expectation zero. If we now define:

$$x = \log x' = X + e$$

$$y = \log y' = Y + f$$

it is seen that the errors in x and y are now uniform, in contrast to the errors in x′ and y′, which are proportional to the values of X' and Y'.

In place of the linear relation $Y' = \alpha' X' + \beta'$, we can now consider the relation $Y = \alpha X + \beta$, and attempt to estimate it by the observed transformed values x, y. Although theoretically, the value of α should be unity, it is more appropriate here to regard it as an unknown parameter with value close to unity, since the various assumptions made may not hold exactly. (For instance, the true relation connecting X', Y' may not be exactly linear, or perhaps the value of β' is not exactly zero, or perhaps the errors in x' and y' are not exactly of the specified form.) Reference to the second graph (figure 6) in which y is plotted against x, indicates that the transformation to logarithms has been effective in making the spread of the errors about the line uniform. Further, it is seen that the slope α is reasonably close to unity.

Variances. Using least squares, the variances of x and y readings, about their

TABLE VII. STATISTICAL ESTIMATES* FOR IBV VALUES

Penetration and soaking time (in.)	(hrs.)	$\Sigma (\bar{x}_i - x_{..})^2$	$4.S_{xyb}$ (y at 0.1 in.)	$\hat{\sigma}_e^2$	$\hat{\sigma}^2$ (y at 0.1 in.)	$\hat{\sigma}^2$ (y at 0.2 in.)	$4.S_{xyb}$ (y at 0.2 in.)
0.06	00	0.3382	0.2336	0.0012	0.0143	0.0035	0.2709
0.06	24	0.9044	0.7991	0.0017	0.0112	0.0225	0.9259
0.06	48	1.5339	1.0219	0.0095	0.0148	0.0221	1.2079
0.06	72	1.8034	1.0831	0.0088	0.0192	0.0365	1.2382
0.08	00	0.3144	0.2000	0.0009	0.0192	0.0052	0.2541
0.08	24	1.0194	0.8419	0.0014	0.0128	0.0219	0.9853
0.08	48	1.6678	1.0532	0.0089	0.0171	0.0235	1.2531
0.08	72	1.7778	1.0872	0.0055	0.0171	0.0304	1.2598
0.10	00	0.2957	0.1703	0.0007	0.0233	0.0071	0.2380
0.10	24	1.0228	0.8395	0.0015	0.0136	0.0208	0.9908
0.10	48	1.7950	1.0819	0.0083	0.0190	0.0248	1.2935
0.10	72	1.6545	1.0604	0.0046	0.0150	0.0246	1.2429
0.16	00	0.3278	....†	0.0006	....†	0.0133	0.2207
0.16	24	1.3014	....	0.0018	....	0.0182	1.1284
0.16	48	1.9074	....	0.0051	....	0.0196	1.3594
0.16	72	1.8459	....	0.0021	....	0.0187	1.3414

*Formulae defining column headings appear in text.

†No lines of relationship were estimated for IBV at 0.16 inch vs. CBR at 0.10 inch penetration.

true values (symbols σ_e and σ_f, respectively) were estimated. A variance may be interpreted as indicating a measure of the spread of observed values about the true value. Thus, for good prediction, low values of σ_e and σ_f are desired. An examination of the estimated values of σ_e^2 for the experimental conditions shown reveals that there is a certain pattern in their behavior (table VII). If, for example, we consider the first four estimated σ_e^2 values (column 3), it is apparent that the variance is lowest for the unsoaked treatment, increases somewhat for the 24 hour soaking period, increases tremendously for the 48 hour soaking, then decreases somewhat for the 72 hour soaking period. The same pattern is repeated for the three sets of four treatments each, corresponding to 0.08, 0.10 and 0.16 inch penetrations.

The homogeneity of the variance corresponding to the first twelve treatments was tested[3]. The results clearly reject the hypothesis that the twelve values of σ_e^2 are equal; we are on relatively safe ground in examining the differences in the values of σ_e^2 for the various conditions of testing.

Due to limitations in available facilities and funds, the main part of the statistical study has been confined to studying the behavior of the first twelve treatments listed in table VII. The four treatments corresponding to 0.16 inch penetration were examined later to ascertain whether the same trend in the values of $\hat{\sigma}_e^2$ persists; and in fact, the corresponding data reveals that this is the case.

A similar analysis was performed on the four CBR treatments listed in table VIII. Examination of the $\hat{\sigma}_f^2$ values reveals that a pattern, analagous

TABLE VIII. STATISTICAL ESTIMATES* FOR CBR VALUES

Penetration and soaking (in.)	time (hrs.)	$\hat{\sigma}_f^2$	$4.S_{yyb}$	$4.S_{yyw}$
0.10	00	0.0009	0.2615	0.0009
0.10	96	0.0052	0.7847	0.0052
0.20	00	0.0009	0.2415	0.0009
0.20	96	0.0025	1.1058	0.0025

*Formulae defining column headings appear in the text.

to that for the IBV treatments, persists. That is to say, the values corresponding to the dry treatments are substantially lower than those corresponding to the 96 hour soaking treatments.

It is apparent from table VII, as well as from table VIII, that the variation in the values of $\hat{\sigma}_e^2$ and $\hat{\sigma}_f^2$ for different depths at the same soaking period is minor as compared to the variation between soaking periods at the same depth. These sources of variation were examined objectively by means of an analysis of variance test applied to the CBR data, and it was

demonstrated that the variation due to depths was insignificant as compared with that of the experimental error, though the variation due to soaking periods was highly significant. A similar analysis for the IBV data was not feasible due to the unequal numbers of readings on different samples; but a similar conclusion that the variation due to depth is insignificant is reasonable.

The relatively minor variation due to depth of penetration and the importance of variation due to soaking periods suggests that, in making predictions of CBR from IBV values, the soaking period, and not the depth of penetration, is of major importance.

Estimation of the Linear Relationships

General. Various methods are used for estimating the parameters in a linear relationship where both variables are subject to error. The ordinary least-squares method leads to biased estimations, since the assumptions underlying a valid application of least-squares include, among others, that the independent variable be free of error. Despite this, the least-squares line can be used for prediction purposes.

Method I[20]. Among the procedures for obtaining estimators which are consistent, (i.e., asymptotically unbiased), Method I assumes that the observations can be separated into two groups according to the values of X, and independently of the errors e. The slope of the line is then determined from the means of the two groups, and the position is determined from the overall mean.

For each IBV determination on a particular soil, three tests were generally performed. For the corresponding CBR determination, only two tests were performed. In attempting to estimate the linear relationship between X and Y, it is therefore appropriate to consider means of the readings for each soil.

Thus, in terms of the transformed variables, X, Y, e, f:

$$x_{ij} = X_i + e_{ij} \qquad i = 1, 2, 3, \ldots, n \qquad j = 1, 2, 3, \ldots, r_1$$
$$y_{ij} = Y_i + f_{ij} \qquad i = 1, 2, 3, \ldots, n \qquad j = 1, 2, 3, \ldots, r_2$$

That is to say, x_{ij} is the observed IBV value corresponding with the jth test on the ith soil; and y_{ij} is the observed CBR value corresponding to the jth test on the ith soil. In these experiments, $n = 9$, $r_1 = 3$, and $r_2 = 2$.

Actually, the value of r_1 was not always exactly three due to limitations in availability of testing equipment. As can be seen in table III, on a few occasions r_1 exceeded three, and a few times it fell below three; but on the average it was equal to three, and with no serious loss of accuracy it will be assumed $r_1 = 3$ throughout.

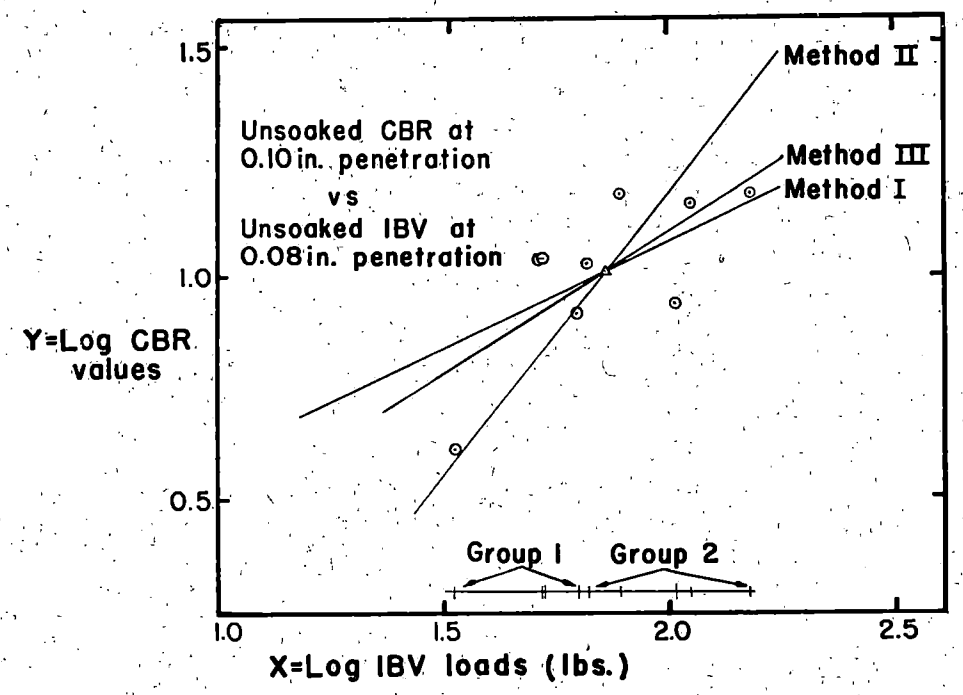

Fig. 5. Fitting line of relationship between typical CBR and IBV values. Lines shown are those estimated by methods I, II, and III.

Let $\overline{x_i}\cdot = \frac{1}{r_1}\sum_{j=1}^{r_1} x_{ij}$, the mean value of the r_1 IBV readings corresponding to the ith soil. Similarly, let $\overline{y_i}\cdot = \frac{1}{r_2}\sum_{j=1}^{r_2} y_{ij}$, the mean of the r_2 CBR readings corresponding to the ith soil.

The points (x_i, y_i) are now used in the estimation of the parameters α, β in the relation $Y = \alpha X + \beta$, using the following formulae:

$$\alpha = \frac{\overline{y}_{(2)}\cdot - \overline{y}_{(1)}\cdot}{\overline{x}_{(2)}\cdot - \overline{x}_{(1)}\cdot} \qquad \beta = \overline{y}\,\ldots - \hat{\alpha}\overline{x}$$

where $\overline{x}_{(2)}$, $\overline{y}_{(2)}$ and $\overline{x}_{(1)}$, $\overline{y}_{(1)}$, represent the means of the upper and lower groups, respectively. These upper and lower groups were obtained simply by dividing the nine observed means $\overline{x_1}\,.\,, \overline{x_2}\,.\,, \ldots, \overline{x_9}\,.$ into two groups of four and five observed points respectively, such that each of the five numbers $\overline{x_1}\,.$ in the second group exceeded all the four numbers $\overline{x_1}\,.$ in the first group. For these two groups, the respective means $\overline{y}_{(2)}$, $\overline{y}_{(1)}$ were computed analogously.

The notation $\overline{x}\,..$ signifies the mean of the nine soil means $\overline{x}_1\,.\,.$, $\overline{x}_2\,.\,.$, $\overline{x}_3\,.\,.$, . . . , $\overline{x}_9$. Similarly, $\overline{y}\,..$ denotes the mean of the nine soil means $\overline{y}_1\,.\,.$, $\overline{y}_2\,.\,.$, $\overline{y}_3\,.\,.$, . . . , $\overline{y}_9$.

The main objection to this method is that the separation of the nine soils into two groups should be effected on the basis of true values X_1, X_2, X_3, . . . , X_9; in practice, we are compelled to separate the soils according to the observed soil means $\overline{x}_i$.

If we now define:

$$\overline{e}_i\,. = \frac{1}{r_1}\sum_{j=1}^{r_1} e_{ij}$$

then, if the errors $\overline{e}_i$ are sufficiently small, the grouping according to the observed $\overline{x}_i$ . . . will be equivalent to the grouping according to the true X_i . . . It frequently happens, however, that the errors $\overline{e}_i$ . . . are not so small; consequently, the grouping is ineffective. In the unsoaked treatments, especially, the errors were not small enough to permit a grouping on the basis of the observed means $\overline{x}_i$ . . . ; this method was therefore abandoned.

The plot of the relationship of a typical set of IBV versus CBR values (transformed to logarithms), shows that the line estimated by Method I does not fit the data as well as does the line estimated by Method II (figure 5). Along the x-axis of this figure are marked the $\overline{x}_i$ . . . values of all observations. From these, it can readily be seen why the fit is so poor. Method I is based on the assumption that the observations can be separated into two distinct groups in order of their x values. From the marks along the x-axis,

TABLE IX. SLOPES OF LINEAR RELATIONSHIP ESTIMATED BY METHODS I, II, AND III

Penetration and soaking time (in.)	(hrs.)	Method I slope (y at 0.1 in.)	Method II slope (y at 0.1 in.)	Method III slope (y at 0.1 in.)	Method II slope (y at 0.2 in.)
0.06	00	0.6284	1.1035	0.6908	0.8780
0.06	24	0.8670	0.9558	0.8836	1.1830
0.06	48	0.7080	0.7475	0.6662	....*
0.06	72	0.6770	0.7053	0.6006	....
0.08	00	0.4905	1.2891	0.6360	0.9360
0.08	24	0.7895	0.9073	0.8259	1.1120
0.08	48	0.6717	0.7253	0.6315	....
0.08	72	0.6960	0.7026	0.6115	....
0.10	00	0.4965	1.5135	0.5760	0.9997
0.10	24	0.7581	0.9098	0.8208	1.1060
0.10	48	0.6213	0.7060	0.6027	....
0.10	72	0.5940	0.7204	0.6409	....
0.16	00	....†	....	....	1.1690
0.16	24	....	....	....	0.9431

*No correlation attempted between 48 and 72 hour soaked IBV and CBR at 0.20 inch penetration, since 0.10 inch CBR studies indicated that 24 hour soaking period gives best predictions. See section entitled "Variances."

†CBR at 0.10 inch penetration not related to IBV at 0.16 inch penetration.

it is seen that the errors $\overline{e_i}$. . . are of greater order than the spacing between consecutive true X values. Indeed, the y values corresponding to several of the points in the lower group are considerably higher than those of several points which, by their x values, fall into the upper group. This is a serious limitation of Method I and seems to rule against its being used. Table IX includes a list of slopes estimated by the method.

Method II. The second method is based on the sums of squares and products of the observed values. The following are consistent estimators of α, β.

$$\hat{\alpha} = \frac{\text{Syyb} - 4\,\text{Syyw}}{\text{Sxyb}} \quad \hat{\beta} = \bar{y}\,.\,. - \hat{\alpha}\bar{x}\,.\,.$$

where

$$\text{Syyb} = \frac{r_2}{n-1} \sum_{j=1}^{n} (\overline{y_i}\,.\,. - \bar{y}\,.\,.)^2$$

$$\text{Syyw} = \frac{1}{n\,(r_2 - 1)} \sum_{1=1}^{n} \sum_{1=1}^{r_f} (y_{ij} - \overline{y_i}\,.\,.)^2$$

$$\text{Sxyb} = \frac{r^2}{n-1} \sum_{i=1}^{n} (\overline{x_i} - \bar{x}\,.\,.)\,(\overline{y_i} - y\,.\,.)$$

Note that the line estimated by method II seems to fit the data quite well (figure 5). Table IX also includes a list of slopes estimated by method II.

Method III (least-squares). Though the estimators obtained by the least-squares method are not consistent, the line estimated by least squares may be used for prediction purposes, by means of confidence bands enclosing the line (figure 7). The least-squares estimators are denoted by the following:

$$\hat{\alpha} = \frac{\sum_{i=1}^{n} (\overline{x_i}\,.\,. - \bar{x}\,.\,.)\,(\overline{y_i} - \bar{y}\,.\,.)}{\sum_{i=1}^{n} (\overline{x_1}\,.\,. - \bar{x}\,.\,.)^2}\,; \quad \beta = \bar{y}\,.\,. - \hat{\alpha}\bar{x}\,.\,.$$

The slopes of the lines computed by methods I, II, and III may be compared (table IX). The slope by method III is less than that by method II. This is to be expected, since the least-squares estimator is not consistent; that of method II is. The bias in estimating the slope due to least-squares estimation in this way should always be downward.

An estimate of the variance of the residuals (deviations) from the least-squares line is given by:

$$\hat{\sigma}^2 = \frac{1}{n-2} \sum_{1=1}^{n} (\overline{y_i}\,.\,. - \hat{\alpha}\overline{x_i} - \hat{\beta})^2$$

Since $\hat{\sigma}^2$ is an estimate of the variance of residuals of means $\overline{y_i}$. from the

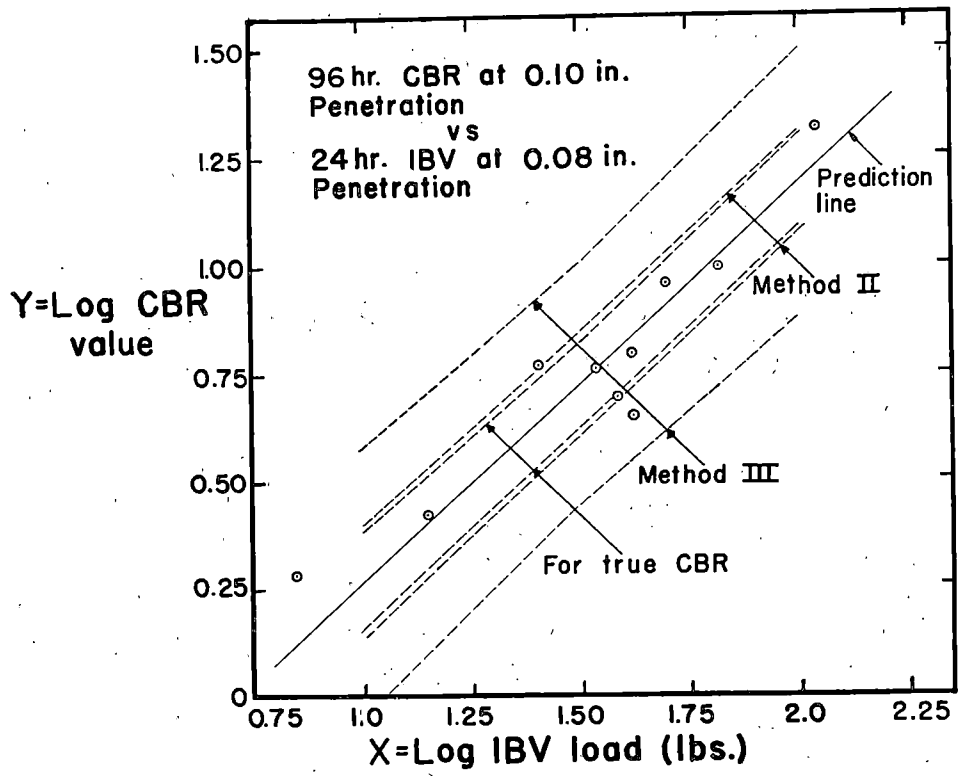

Fig. 6. Prediction of CBR from three IBV tests. Dashed curves shown limits of 95 percent confidence bands estimated by methods II and III, and by modified method for prediction of true CBR. Prediction line is the line of relationship estimated by method II.

regression line, therefore $\hat{\sigma}^2$ will be 1/r times the variance of residuals of single readings y_{ij} from the same regression line. From this, it is seen that σ^2 is directly proportional to $1/_r$. It makes possible a simple method of altering confidence limits to allow for a change in the number of IBV readings used in prediction. The estimate for σ^2, above, will be used with the formation of a confidence band about the least-squares line.

Prediction of CBR from IBV

General. Suppose a soil sample is to be tested for its IBV load at a given penetration and soaking condition, and its corresponding CBR value is to be predicted. This soil sample, of course, is distinct from and independent of the samples which have been used in computing the estimated line, say by method II or III. For the new soil sample, let x be the IBV obtained as an average of r_1 readings, and y be the corresponding CBR value, which is an average of r_2 readings.

Method II. The limits of the confidence band estimated by Method II are as follows:

$$y = \hat{\alpha}x + \hat{\beta} \pm 1.96 \sqrt{\left[\frac{\hat{\sigma_f}^2}{r_2} + \frac{\hat{\alpha}^2 \hat{\sigma_e}^2}{r_1}\right]\left[\frac{n+1}{n} + \frac{(x - \bar{x}\,..)^2 + \frac{\hat{\sigma_e}^2}{nr_1}}{\Sigma\,(\bar{x}_j\,. - \bar{x}\,..) - (n-1)\frac{\sigma_e^2}{r_1}}\right]}$$

1.96 is obtained from the normal integral.

It can be seen that this formula is that of a hyperbola, whose transverse axis is coincident with the line of relationship estimated by method II. In the plot of these limits for another typical set of data not all of the observed points are enclosed (figure 6).

The undue narrowness of this confidence band may be explained by the following: First, the formula expressing the confidence limits is based on large sample theory, that is to say, the true values of α, σ_e, σ_f, appearing under the square root sign, are replaced by estimates thereof. Second, the variances of σ_e^2, σ_f^2 are apparently small enough to detect a lack of exact linearity in the relationship connecting X and Y. Third, due to the fact that the same soil specimen is used for all penetrations corresponding to a given soaking treatment, the selection is not completely random; hence, the estimates $\hat{\sigma_e}^2$, $\hat{\sigma_f}^2$ will tend to be biased downward.

In view of the above, although the linear relationship estimated by method II is satisfactory, the confidence band, for reason stated, is too narrow.

Method III. The limits of the 95 percent confidence band estimated by method III, the least-squares method, are as follow:

$$y = \hat{\alpha}x + \hat{\beta} \pm 2.36 \sqrt{\sigma^2\left[\frac{n-1}{n} + \frac{(x - \bar{x})^2}{\Sigma\,(\bar{x}_j - x\,..)^2}\right]}$$

2.36 is obtained from tables of the t distribution.

This, again, represents a hyperbola, whose transverse axis coincides with the line of relationship estimated by least-squares. However, as expected, these limits are excessive — that is, they more than adequately enclose all of the observed points (figure 6).

Compromise. The approximately oriented method III confidence band is actually plotted about the method II line; however, the width of the band measured in a vertical direction would be the same about the method III line. Since method II gives the better linear relationship, and since method III gives more than sufficiently wide confidence bands, a compromise is now in order, using the method II line of relationship and the method III confidence band. Since the least-squares confidence band must be wider to compensate for a biased line, this band, when properly oriented about the

method II line, should provide conservative estimates of the predicted values. Hence, the confidence coefficient for this new band should be at least 95%. All observed points fall easily within the limits of the modified confidence band (figure 6).

Modification to predict true CBR. The above confidence bands are based on performing exactly r_1 IBV tests and exactly r_2 CBR tests. If more tests are performed, that is, if r_1 and r_2 are increased, $\frac{\sigma_f^2}{r_2}$ and $\frac{\sigma_e^2}{r_1}$ will decrease. Suppose we are not interested in predicting the CBR value obtained by 2 CBR readings, but rather as usually is the case, we are interested in the *true* CBR value or, in other words, the value which we would obtain by performing an infinite number of CBR tests. Since the confidence bands must take into account errors in results of both the IBV and CBR tests, and since we know that an infinite number of CBR tests would have zero expected error, then it should be possible to reduce the width of the confidence bands, if we are presumably predicting this *true* CBR.

Careful comparison of the formulae for the method II and method III confidence bands suggests a method by which these confidence bands might logically be narrowed. Since the computed quantities $\frac{\hat{\sigma}_e^2}{nr_1}$ and $(n-1)\frac{\hat{\sigma}_e^2}{r_1}$ are small, the only difference in the terms under the radicals of the two formulae is that in the method II formula the quantity $\frac{\hat{\sigma}_e^2}{r_2} + \frac{\hat{\alpha}^2\hat{\sigma}_e^2}{r_1}$ replaces the $\hat{\sigma}^2$ which appears in the method III formula. Though the formula for the confidence bands by method I is not given, it too manifests a very similar likeness to the method III formula. By analogy between the formulae, it seems reasonable to assume that the method III $\hat{\sigma}^2$ can be broken down into 2 parts: one due to the variance of the IBV values, and the other due to the variance of the CBR values. By this reasoning, the following ratio seems warranted:

$$\frac{\hat{\sigma}^2 \text{ of method III}}{\hat{\sigma}^2 \text{ with } r_2 = \infty} = \frac{\frac{\hat{\sigma}_f^2}{r_2} + \frac{\hat{\alpha}^2\hat{\sigma}_e^2}{r_1}}{\frac{\hat{\alpha}^2\hat{\sigma}_e^2}{r_1}}$$

Using this new value of $\hat{\sigma}^2$ in the method III formula for confidence bands yields the limits shown (figure 6). These confidence limits, based on r_1 IBV readings, enclose the true CBR values with a probability exceeding 95 percent.

Choice of best prediction lines. The next step is to choose the four linear relationships which give lowest errors for predicting CBRs in both the soaked

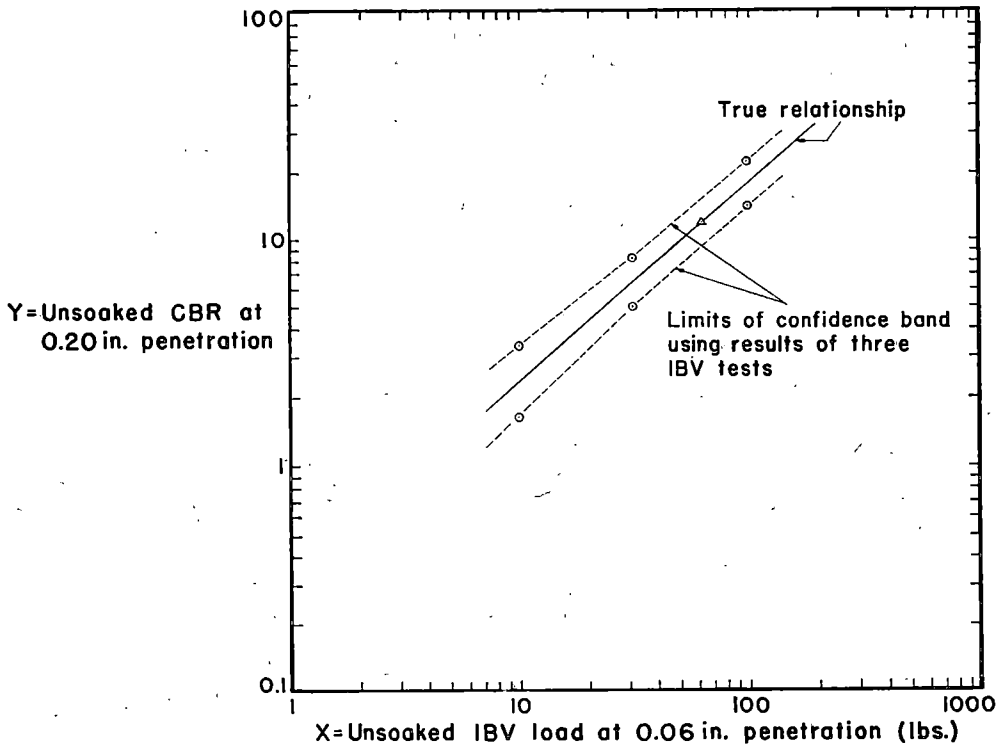

Fig. 7. Curve for prediction of unsoaked CBR at 0.10 inch penetration. Enter graph at x value corresponding to average IBV reading. Read up to the intersection of this x value and the "true relationship" line. The true CBR is the y value of this point of intersection.

and unsoaked tests, and at both 0.10 and 0.20 inch penetrations. To make this choice, the lines represented by each of the slopes listed in table IX were plotted.

The confidence bands were estimated both by least-squares and by the modified method described above for each of these lines. It was then a simple matter to choose the line which gives the lowest expected error, as defined by these confidence bands. By this procedure, the following lines were chosen:

For predicting CBR at 0.10 inch penetration:

Unsoaked: Use unsoaked IBV at 0.06 inch penetration.

$$Y = 1.1035\,X - 0.9675$$

Soaked: Use 24 hour soaked IBV at 0.08 inch penetration.

$$Y = 0.9073\,X - 0.6287$$

For predicting CBR at 0.20 inch penetration:

Unsoaked: Use unsoaked IBV at 0.06 inch penetration.

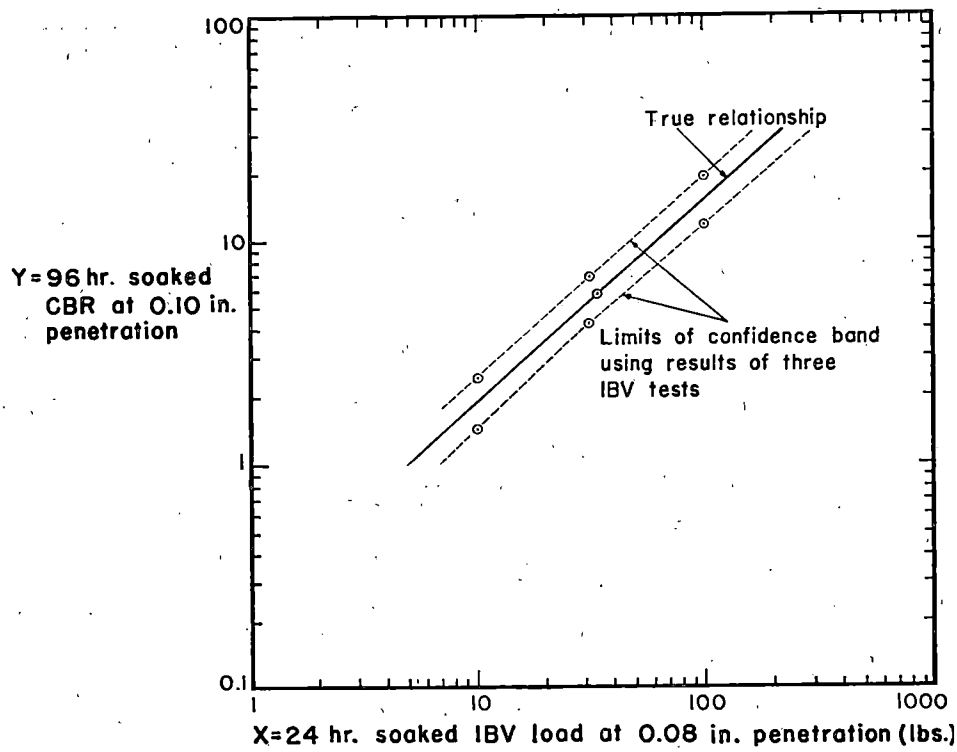

Fig. 8. Curve for prediction of 96 hour soaked CBR at 0.10 inch penetration. Enter graph at x value corresponding to average IBV reading. Read up to the intersection of this x value and the "true relationship" line. The true CBR is the y value of this point of intersection.

$$Y = 0.8780\ X - 0.5160$$

Soaked: Use 24 hour soaked IBV at 0.10 inch penetration.

$$Y = 1.1058\ X - 0.9737$$

These four lines are plotted on a logarithmic scale (figures 7, 8, 9, and 10). Theoretically, to use these curves one should first find the logarithm of each of the indicated IBV load values, then average these logarithms and take the antilog of this average. This antilog is the abscissa to use on the appropriate graph in prediction. However, due to the relatively small differences between results of successive IBV tests, little error is introduced by merely taking the average of the test results as the abscissa on the appropriate graph.

Although the above choices of lines indicates particular combinations of penetrations and soaking periods, it is considered unlikely that the choice of slightly different penetrations would affect seriously the accuracy of prediction. The reason for this is apparent from the remarks regarding the minor role played by the variations due to penetrations. This was borne out

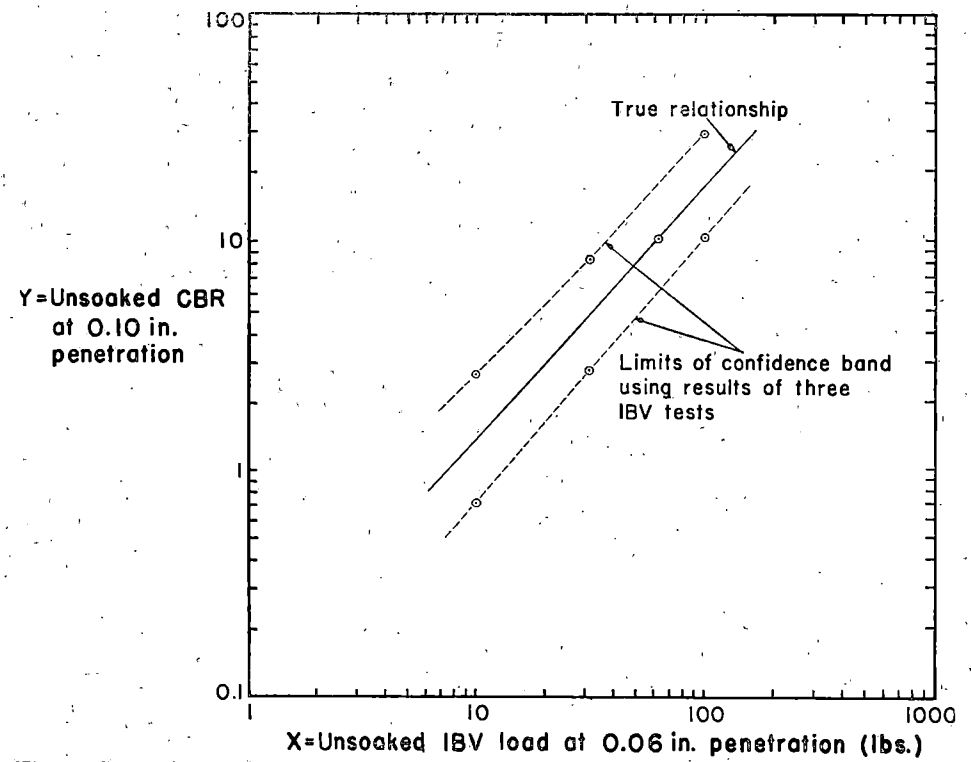

Fig. 9. Curve for prediction of unsoaked CBR at 0.20 inch penetration. Enter graph at x value corresponding to average IBV reading. Read up to the intersection of this x value and the "true relationship" line. The true CBR is the y value of this point of intersection.

by an examination of the confidence bands estimated for different penetrations at the same soaking period.

Reducing confidence bands by increasing number of IBV tests. We will now examine the effect which the number of tests used for a prediction has on the width of corresponding bands. In accordance with the "Prediction of CBR from IBV" above the formula for confidence bands, for at least 95 percent confidence coefficient, is as follows:

$$y = \alpha x + \beta \pm 2.36 \sqrt{\frac{\frac{\hat{\alpha}^2\hat{\sigma}_e^2}{r_1} \quad \hat{\sigma}^2}{\frac{\hat{\sigma}_f^2}{r_2} + \frac{\hat{\alpha}^2\hat{\sigma}_f^2}{r_1}} \left[\frac{D+1}{n} + \frac{(x-\bar{x})^2}{\Sigma\,(x_i . - x . .)^2}\right]}$$

Substituting appropriate values for the relationship: 96 hour soaked CBR at 0.10 inch versus 24 hour soaked IBV at 0.08 inch, the formula becomes:

$$y = 0.9073x - 0.6787 \pm 2.36\,(0.0411)\sqrt{1.1111 + \frac{(x-1.5309)^2}{1.0194}}$$

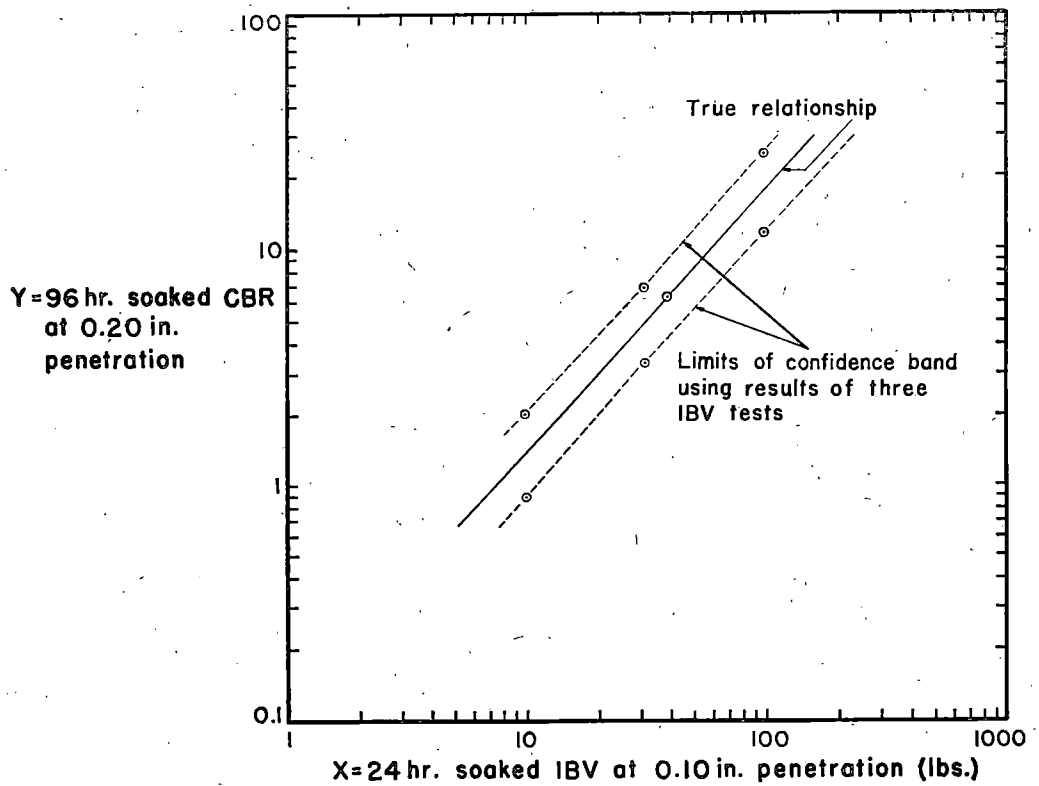

Fig. 10. Curve for prediction of 96 hour soaked CBR at 0.20 inch penetration. Enter graph at x value corresponding to average IBV reading. Read up to the intersection of this x value and the "true relationship" line. The true CBR is the y value of this point of intersection.

Careful examination of this formula indicates that the factor to the right of the $\pm$ sign is equal to half the magnitude of the y interval of the confidence band for a given value of x.

The above formula is the type used to compute the confidence bands plotted as dashed curves (figures 7, 8, 9, and 10). Actually it was necessary to convert back to antilogs for these graphs to be able to plot the confidence bands on the logarithmic graph paper used.

Now suppose that instead of using r_1 in the IBV tests, we wish to use r_1'. In the formula for the confidence bands, the original r_1 and r_2 remain the same, since these relate to the original data used in formulating the linear relationship. However, it is known that $\hat{\sigma}^2$ varies inversely as r. Therefore, for a new number of IBV tests, r_1', we multiply $\hat{\sigma}^2$ by the factor r_1/r_1', and the y interval enclosed by the confidence band is multiplied by the factor $\sqrt{r_1/r_1'}$.

Thus, if we wish to reduce the expected error of prediction by using

nine IBV tests instead of three, we simply multiply the y interval between the confidence limits by a factor of $\sqrt{3/9} = \sqrt{1/3}$.

The accuracy of predicting CBR from IBV by use of the linear relationship of figures 7 through 10 is limited only by the number of IBV tests which are used in the prediction. If greater accuracy is desired, a larger number of IBV tests must be made. The 95 percent confidence intervals, based on using three IBV tests in prediction, are as plotted on these four prediction graphs. These provide sufficient accuracy for most routine laboratory investigations.

CONCLUSIONS

1. The Iowa Bearing Value test, in the form recommended in the Appendix of this report, is a satisfactory test for prediction of California Bearing Ratios for soils of the types studied. The more important recommendations concerning procedure for performing the IBV test for making the prediction are as follows:

a. The best linear relationships for prediction of the CBR at various conditions are as follows:

For predicting the unsoaked CBR at 0.10 inch penetration, use the unsoaked IBV load read at 0.06 penetration. The linear relationship is $Y = 1.1033X - 0.9673$, where Y is the average logarithm of CBR values and X is the average logarithm of IBV values (figure 7).

For predicting the soaked CBR at 0.10 inch penetration, use the 24 hour soaked IBV load read at 0.08 inch penetration. The linear relationship is $Y = 0.9073X - 0.6278$ (figure 8).

For predicting the unsoaked CBR at 0.20 inch penetration, use the unsoaked IBV load read at 0.06 inch penetration. The linear relationship is $Y \pm 0.8780X - 0.5160$ (figure 9).

For predicting the soaked CBR at 0.20 inch penetration, use the 24 hour soaked IBV load read at 0.10 inch penetration. The linear relationship is $Y = 1.1058X - 0.9737$ (figure 10).

b. Results of the 24 hour soaked IBV test correlate better with 96 hour soaked CBR test results than do the results of either the 48 or 72 hour soaked IBV tests.

c. In performing the IBV test, it is not necessary to penetrate the specimen to a depth greater than 0.20 inch.

d. Confidence bands for at least 95 percent prediction probability can be estimated by the method outlined in the Statistical Analysis section above.

The exact relationship between the reliability of prediction and the number of IBV tests used for the prediction is as indicated under reducing confidence bands by increasing number of IBV tests.

2. Expansion data obtained from the nine soil samples used in this study

are not considered adequate to make an exact correlation between the expansion characteristics of the IBV and CBR specimens. However, results of the IBV expansion test do give useful information on the relative tendency of soils to swell when immersed in water.

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APPENDIX

RECOMMENDED PROCEDURE FOR PERFORMANCE OF THE IOWA BEARING VALUE TEST

Scope

This method of test, as herein outlined, is intended to determine the following:

1. The relative bearing capacity of soils compacted to near standard Proctor density (ASTM designation: D698-42T). The bearing capacity of the specimen can be evaluated in the "as molded" condition and/or after any subsequent treatment such as immersion in water, air-drying, freezing-and-thawing, etc.
2. The extent to which subgrade or base materials will swell and absorb water during a soaking period of 24 hours or any other desired soaking period.
3. The estimated California Bearing Ratio of the soil from a given number of IBV tests performed as indicated below.

Apparatus

The apparatus for performing the IBV test is the following (figure 1):

Cylindrical mold. A cylindrical mold is 2 in. in inside diameter and 5 inches in height. The mold shall be fitted with a steel penetration piston ⅜ inch in diameter, mounted to slide in a brass guide which fits as a cover for the cylinder during the actual penetration test.

Molding machine. The molding machine used for preparing the IBV specimen consists of a steel pedestal on a steel stand, above which is mounted a steel yoke which serves as a guide for the 5 pound drop hammer. The hammer slides on a steel shaft which is inserted into the upper end of the cylindrical mold. At its upper end the hammer shaft fits into a guide slot in the yoke. The hammer drops 12 inches, striking a cap attached to the lower end of the shaft. This cap bears against the soil in the mold. The machine is also equipped with a lever for forcing the cylindrical mold down over the pedestal after the compaction is complete. This allows the excess soil to be struck off with a straight-edged blade, insuring a test specimen exactly 2 inches high.

Soaking frame. The soaking frame is a brass platform on 3 or 4 short legs which allow water to circulate under the entire soaking assembly. Perforations in the base of the platform allow entry of water into the lower end of the specimen. The frame has a top hold-down ring which is tightened against the platform by means of two bolts and wing nuts. The top ring is recessed slightly to allow an easy, plumb fit of the mold (which contains the specimen during soaking) inside the frames. The top surface of the hold-down

ring is accurately machined to allow an exact, unchanging fit between this device and the stand of the expansion gage device.

Scales. A scale or balance of 750 gram capacity, sensitive to 1 gram, is used.

Dials. A dial or other suitable gage is used for measuring the penetration of the $\frac{5}{8}$ inch piston. A second dial or gage is mounted on the expansion gage device.

Water tank. A suitable water tank or vat is necessary for immersing the specimens during the soaking period.

Drying oven. A suitable drying oven, with forced ventilation, is used for determining moisture contents.

Miscellaneous apparatus. Mixing bowl, spatulas, a straight-edged striking off blade, spoons, etc. are also needed.

Sample

A representative sample of sufficient size (about 200 grams required per specimen) is air dried, then separated by screening through the No. 10 sieve. Only material passing the No. 10 sieve is used in preparing the test specimen.

Compacted Test Specimen

The sample is mixed at optimum moisture content, and about 200 grams is weighed out in the preparation of one specimen. The procedure for preparing one specimen is as follows:

1. Weigh out enough moist soil to make a specimen slightly over 2 inches high after compaction.
2. Pour the moist soil into the mold, which rests over the 3 inch pedestal and is supported on temporary supports. The mold is fitted with a brass collar which prevents the loose soil from overflowing the apparatus.
3. Insert the hammer into the upper end of the mold; then raise and drop the hammer weight once in order to give the specimen enough lateral pressure so that the temporary supports may be removed.
4. Remove the temporary supports, and give the specimen 4 more blows of the hammer.
5. Remove the collar and invert the mold. The top of the partially compacted specimen in the mold becomes the bottom and is in contact with the top surface of the 3 inch pedestal.
6. Complete the compaction by raising and dropping the hammer 5 more times. This compactive effort is equivalent to that used in standard Proctor compaction.
7. With the compacted specimen in the mold and resting on the pedestal, push the mold downward, using the extruding lever device, until the lower end of the mold contacts the base of the pedestal. Since the mold is 3 inches high and the pedestal 5 inches high, the portion of the specimen in excess

of 2 inches in height is thereby pushed out of the mold. Strike off the excess soil to make the specimen exactly 2 inches high.
8. Weigh the specimen and take a sample of the unused soil for moisture content determination. The dry density of the specimen can be computed, since the exact dimensions of the specimen and its weight are known, and its moisture content can be determined.
9. After weighing, the sample can be used either for immediate "as molded" penetration testing, or can be placed in the soaking frame and immersed in water before the penetration test.

Penetration Test

The compacted specimen in the mold is placed in the testing machine and tested by penetrating it with the $\frac{5}{8}$ inch piston. Before starting the penetration test, the mold cover-piston guide device is placed over the mold and the piston inserted and lowered carefully until its lower end rests on the upper surface of the specimen. The penetration gage is then calibrated either mechanically by adjusting the dial reading to zero, or by simply recording the registered reading, which is the zero penetration reading to be subtracted from later readings. During the penetration test, the head of the testing machine is operated at the rate of 0.05 inch per minute, and the total load in pounds is recorded at penetrations of 0.02, 0.04, 0.06, 0.08, 0.10, 0.12, 0.14, 0.16, 0.18, and 0.20 inch.

Expansion

The IBV expansion test is performed on prepared specimens as follows:
1. The mold, containing this specimen, is inserted into the soaking frame. The upper and lower surfaces of the soil are covered with a single layer of coarse filter paper. The filter paper at the lower surface acts as a porous membrane between the soil and the perforated base of the soaking frame; the paper at the upper surface separates the soil and the surcharge weight device which rests on the soil during soaking. The stem of the surcharge weight device is of the correct length for measurement of specimen height changes by means of the expansion gage device. After the entire apparatus —mold plus soaking frame and surcharge weight device—is assembled, it is immersed in a tank of water which is of such depth that the water surface is about 1 inch over the top edge of the cylindrical mold.
2. The specimen is soaked for a period of 24 hours. Expansion readings are taken immediately after immersion and just prior to removing the specimen from the water.
3. After the 24 hour soaking period, the specimen is removed from the tank and with surcharge weight device held in place the mold is tipped up, allowing excess water to run out of the top of the mold. Then the surcharge weight device and filter paper are removed and the specimen inverted and

allowed to drain for a period of 5 minutes.

4. After draining, the mold is removed from the soaking frame, and the filter paper is stripped off the lower surface of the specimen. Then the soaked specimen plus mold is weighed to determine the increase in weight due to absorption.

Penetration Test of Soaked Specimen

The soaked specimen is tested for penetration resistance as specified above for the unsoaked specimen.

Moisture Content

After any penetration test, the specimen is removed from the mold and a moisture content determination is made of a sample cut from the specimen. The moisture sample is dried to constant weight in accordance with the standard moisture content determination procedure. The calculated moisture content is recorded.

The specimen may be extruded from the cylindrical mold by simply pushing the mold down over a metal rod slightly less than 2 inches in diameter and at least 5 inches high. If the specimen is then sliced as one would slice a pie, a good representation of the entire sample is obtained for moisture determination.

Prediction of California Bearing Ratio

If it is desired to predict the CBR value of the soil from IBV results, this can be done as follows:

1. To predict the unsoaked CBR at 0.10 inch penetration, enter the graph, figure 9, at the x value corresponding to the average unsoaked IBV load at 0.06 inch penetration. Read up to the intersection of this abscissa and the correlation line, then across to the y intercept corresponding to this intersection. This y value is the CBR of the soil. If an average of the loads of three IBV tests is used, the maximum probable error of prediction (95 percent probability) will fall within the confidence bands whose boundaries are indicated by the dashed curves on the appropriate graph.

2. To predict the 96 hour soaked CBR at 0.10 inch penetration enter the graph, figure 10, at the x value corresponding to the average 24 hour soaked IBV load at 0.08 inch penetration. From here, follow instructions in 1, above, which begin "Read up to the intersection . . . ".

3. To predict the unsoaked CBR at 0.20 inch penetration, enter the graph, figure 11, at the x value corresponding to the average unsoaked IBV load at 0.06 inch penetration. From here, follow instructions in 1, above, which begin "Read up to the intersection . . . ".

4. To predict the 96 hour CBR at 0.20 inch penetration enter the graph, figure 12, at the x value corresponding to the average 24 hour soaked IBV load at 0.10 inch penetration. From here, follow instructions in 1, above,

which begin "Read up to the intersection . . . ".

If it is desired to determine the confidence bands for prediction using more or fewer than two IBV tests, these can be estimated as in "Statistical Analysis," above.

Determination of Percent Expansion

Percent expansion is determined by dividing the total expansion (in inches) after 24 hours of soaking by the original height of the specimen (2 inches). This is a method of obtaining an estimate of the expansion to be expected under field conditions. It has several obvious weaknesses. For example, expansion during this test is somewhat restrained due to friction between the soil and the rigid walls of the cylindrical mold. Therefore, expansion data obtained by this method should be supplemented by data from other expansion tests. Several are in common use in most testing laboratories. However, the percent expansion obtained by this method does rate the tendency of the soil to expand on soaking, and can be used as such.

CORRELATION OF THE CALIFORNIA BEARING RATIO AND THE IOWA BEARING VALUE

by

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(Progress Report, 1957)

ABSTRACT

Curves for the prediction of the California Bearing Ratio of sandy, silty, and clayey soils from the more easily and quickly obtained Iowa Bearing Value are presented. A statistical approach is used in the correlation.

INTRODUCTION

The present road building programs require that many investigations of the bearing capacity of sub-grade, sub-base, and base course materials be made in a short time. A rapid method for determining the bearing value of materials is needed. The Iowa Bearing Value test, developed by the Iowa Engineering Experiment Station under the sponsorship of the Iowa State Highway Commission, is a rapid method for determining the bearing value of sandy, silty and clayey soils.

A widely used method for determining the necessary thickness of base materials for flexible pavements uses the California Bearing Ratio. The U. S. Army Corps of Engineers[11] has adopted the CBR method of design for airfields as well as roads. Experience with the method has proved that it is sound. However, the testing procedure for determining the CBR of a soil has several disadvantages. A relatively large soil sample is necessary. At least two persons are required to conduct the test. The testing procedure requires an excessive amount of time. The IBV test was developed to overcome these disadvantages.

The IBV test is somewhat similar to the CBR test (figure 8). The specimen is compacted into a two inch diameter mold and struck off to a height of two inches. The specimen remains in the mold during testing with a five-eighths inch diameter penetration rod. The test requires one-twentieth of the amount of material required for the CBR test and can be made by one man. The test takes less than half the time of the CBR test, and the smaller specimen requires a shorter soaking period than for the large CBR specimen. In its present state of development the IBV test is used with materials finer than the No. 10 sieve which do not contain more than 80 percent of sand. A

limited amount of research indicates that materials containing up to 25 percent gravel of one-quarter inch maximum size also may be tested[2].

Since the CBR design curves have been service correlated, it was decided to correlate the IBV with the CBR to take advantage of these design curves. Curves correlating the IBV and the CBR of natural soils and soil-aggregate mixtures were developed[2, 5].

Though the accuracy of curves for higher CBR values is not as great as desirable, a relation between the variations in density of the IBV specimens from that of the CBR specimens and the sand content of the material was determined[2]. A continuation of this investigation showed that more accurate predictions of the CBR could be made if separate curves were developed for two groups of materials, one with less than fifty percent sand, and the other with greater than fifty but less than eighty percent sand. The curves developed enable the prediction of the CBR based on the IBV and a routine mechanical analysis.

MATERIALS AND METHODS

The soils selected for this study are all from Iowa. The selection was made to give a sample from as many of the soil textural classes as possible.

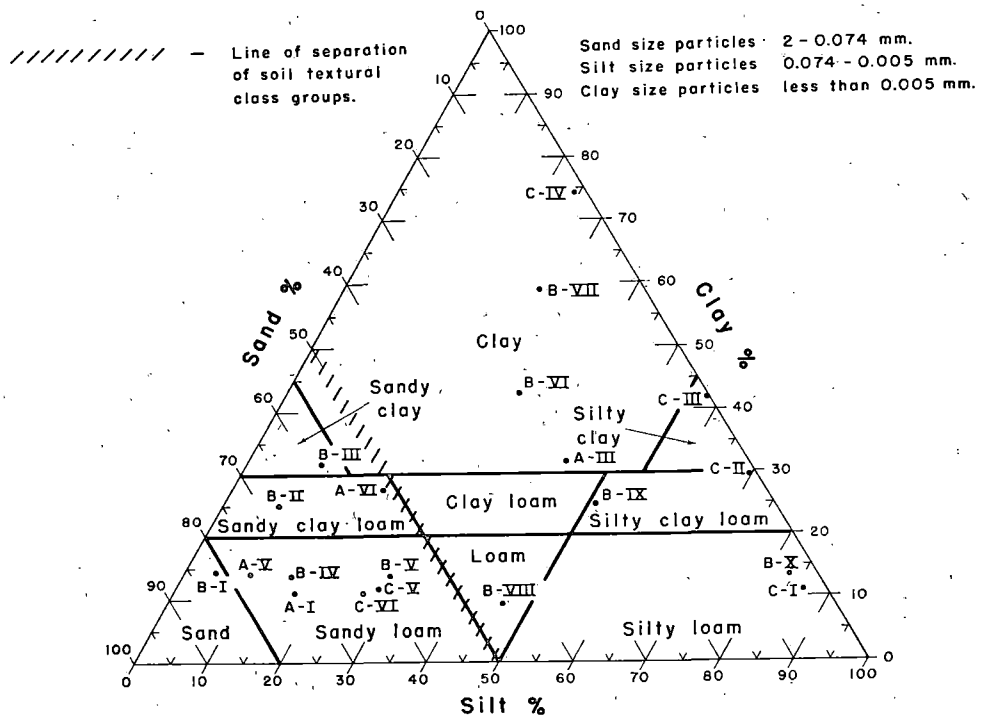

Fig. 1. Distribution on textural classification chart of soils tested.

Mixtures of available soils were made when the natural soils did not have the desired gradation (figure 1 and table I).

TABLE I. PROPERTIES OF SOILS

Sample Number	Properties: Mech. Analysis* % sand	% silt	% clay	O.M.C. % Standard Proctor	Modified AASHO	Textural Classification†
A-1	72.5	16.7	10.8	.....	7.2	Sandy loam
A-II	24.7	43.5	31.8	.....	16.4	Clay
A-V	76.5	9.5	14.0	9.4	.....	Sandy loam
A-VI	52.0	20.8	24.2	12.8	.....	Sandy clay loam
B-II	67.3	7.8	24.9	11.5	10.0	Sandy clay loam
B-III	58.0	10.1	31.9	14.5	12.5	Sandy clay
B-IV	71.8	15.0	13.2	10.5	9.5	Sandy loam
B-V	58.2	28.6	13.2	12.0	10.5	Sandy loam
B-VI	25.3	32.1	42.6	15.5	12.8	Clay
B-VII	14.1	26.8	59.1	19.0	16.0	Clay
B-VIII	45.0	46.0	9.0	9.0	8.0	Loam
B-IX	24.2	51.0	24.8	12.5	10.0	Silty clay loam
B-X	3.6	83.2	13.2	16.5	14.0	Silty loam
C-I	3.0	86.0	11.0	15.5	13.0	Silty loam
C-II	0.9	69.7	29.4	17.7	11.6	Silty clay
C-III	0.2	58.0	41.8	19.5	15.8	Silty clay
C-IV	1.5	24.2	74.3	23.0	19.1	Clay
C-V	60.5	28.0	11.5	10.4	.....	Sandy loam
C-VI	63.0	26.1	10.9	9.8	.....	Sandy loam

*Sand size particles are retained on the No. 200 sieve; Silt size particles pass the No. 200 sieve and are larger than 5 microns; and clay size particles are smaller than 5 microns.
†(Figure 1).

To attain high CBR values as well as low ones, CBR and IBV specimens were molded at both standard Proctor density and modified AASHO density[1] for most of the soils.

Four standard CBR specimens and six two inch diameter by two inch high IBV specimens were molded for each test at the optimum moisture content for the desired density. Two of CBR specimens and three of the IBV specimens were tested immediately after molding. The other two CBR specimens were immersed in water for a period of four days before testing, and the other three IBV specimens were immersed for a period of two days before testing.

The CBR tests were performed in accordance with the procedure set forth in ASTM manual "Procedure for Testing Soils"[1] with the following modifications:

1. Specimens were compacted at the proper optimum moisture content to either standard Proctor density or modified AASHO density.
2. The immersion time for soaked specimens was four days, and the surcharge weight during immersion was 10 pounds.

3. No surcharge was used during the penetration test. A preliminary study showed that use of comparable surcharges on CBR and IBV specimens during the penetration test does not significantly affect correlation data.

4. Bearing ratios were computed at 0.1 inch and 0.2 inch penetration. The IBV tests were performed in the manner set forth in Appendix A. The results of these tests were analyzed statistically (Appendix B).

This analysis was performed on the logarithms of the CBR and IBV values for reasons pointed out by Lafleur and Arpacioglu. From the statistical analysis the selection was made of the penetration at which the IBV is most closely related to the CBR. The slopes and intercepts of the regression lines were then determined. Finally the regression lines were plotted on log-log paper for use in predicting the CBR.

TABLE II. RESULTS OF CBR AND IBV TESTS (UNSOAKED)

Sample Number	CBR		IBV			
	0.1″	0.2″	0.04″	0.06″	0.08″	0.10″
A-I	122	122	402	522	613	693
A-V	28.3	28.6	163	198	230	260
A-VI	16.0	14.7	96	110	121	127
B-II	11.9	14.7	82	98	106	125
B-II	41.1	54.4	226	281	314	340
B-III	9.2	8.6	47	54	58	63
B-III	22.1	21.0	138	152	167	181
B-IV	2.4	3.0	19	27	37	44
B-IV	3.4	4.2	24	38	52	68
B-V	5.3	6.1	24	34	46	55
B-V	5.8	8.9	55	85	109	139
C-V	4.1	5.3	15	25	34	44
C-V	15.4	20.6	98	127	152	177
A-III	79.0	68.9	355	412	452	474
B-VI	20.3	17.7	65	76	88	93
B-VI	77.2	64.6	255	281	317	336
B-VII	12.2	12.2	62	73	79	89
B-VII	58.2	48.5	220	228	239	267
B-VIII	66.6	64.7	79	90	110	119
B-VIII	150	167	382	467	490	
B-IX	35.4	42.8	81	101	121	139
B-IX	138	156	315	395	502	532
B-X	35.9	41.2	65	91	99	120
B-X	93.0	111	262	320	363	413
C-I	8.4	11.2	47	62	77	91
C-I	87.4	106	278	335	367	355
C-II	10.7	10.9	48	59	67	72
C-II	77.6	70.1	368	433	488	535
C-III	11.1	9.6	42	48	52	56
C-III	47.0	39.0	238	265	287	302
C-IV	11.2	10.7	42	48	54	62
C-IV	45.1	37.3	160	177	190	198

TABLE III. RESULTS OF CBR AND IBV TESTS (SOAKED)

Sample Number	CBR		IBV			
	0.1″	0.2″	0.04″	0.06″	0.08″	0.10″
A-I	80.5	83.5	365	448	510	565
A-V	14.2	12.1	60	70	74	78
A-VI	2.4	2.5	39	42	46	48
B-II	7.4	9.5	66	72	77	95
B-II	17.0	18.2	39	57	80	85
B-III	5.6	5.1	16	16	20	24
B-III	6.5	6.6	15	19	24	27
B-IV	3.0	4.6	46	56	79	88
B-IV	3.4	5.3	31	62	98	127
B-V	3.2	3.8	13	17	21	25
B-V	4.4	5.6	30	40	54	66
C-V	4.5	5.8	23	35	46	55
C-VI	20.6	27.1	81	102	122	139
A-III	3.1	3.1	12	16	18	21
B-VI	1.3	1.1	7	7	9	8
B-VI	3.2	3.0	8	9	9	11
B-VII	1.0	0.8	7	8	9	9
B-VII	2.0	1.8	7	9	9	10
B-VIII	2.6	3.6	8	10	15	15
B-VIII	32.4	40.8	84	74	95	101
B-IX	1.8	1.6	6	5	12	14
B-IX	46.2	52.0	88	91	93	115
B-X	18.5	26.6	12	13	17	36
B-X	38.4	53.6	77	153	147	183
C-I	5.9	7.9	19	24	28	32
C-I	67.7	70.5	111	122	128	138
C-II	6.3	6.1	28	32	38	43
C-II	2.5	3.0	8	10	12	12
C-III	5.0	4.3	28	32	34	36
C-III	6.7	6.8	17	21	26	31
C-IV	1.8	1.5	3	3	4	4
C-IV	1.0	1.1	13	15	15	16

RESULTS

The lower and upper limits of IBV penetration for use in the statistical analysis were set at 0.04 inch and 0.20 inch respectively. The lower limit was chosen because surface irregularities in the specimen at lower penetrations lead to erratic results. The upper limit was chosen because many specimens show evidence of progressive shear failure at greater penetrations. This condition results in increasing penetration with an unchanging or decreasing load (tables II and III).

In both of the previous correlations[2, 5] variations of the IBV soaking periods were studied, and two soaking periods were selected for prediction of CBR. This doubled the number of IBV specimens necessary for the prediction of the soaked CBR. In the continuation study a single IBV soaking period that would give good results in predicting the CBR at either of two penetrations was desired. The two day IBV soaking period selected for this

study does give good results in predicting the CBR, and specimens soaked for two days absorb water more uniformly than those soaked for a shorter period.

The statistical analysis showed that for the prediction of the CBR of unsoaked specimens the IBV at 0.08 inch penetration gives the best results. For the prediction of the CBR of soaked specimens the IBV of 0.10 inch penetration gives the best results. The IBV penetration to be used is independent of the percent of sand in the material and of the penetration at which the CBR is desired. But separate curves for the prediction of the CBR at each of the penetrations studied and for each of the two soil textural class groups studied were necessary.

The equations of the curves for the prediction of the CBR from the IBV of a soil are as follows:

Unsoaked CBR at 0.1 inch penetration
Under 50% sand $Y = 1.3151X - 1.6015$
50% to 80% sand $Y = 1.2333X - 1.1741$

Unsoaked CBR at 0.2 inch penetration
Under 50% sand $Y = 1.2234X - 1.3567$
50% to 80% sand $Y = 1.2807X - 1.2758$

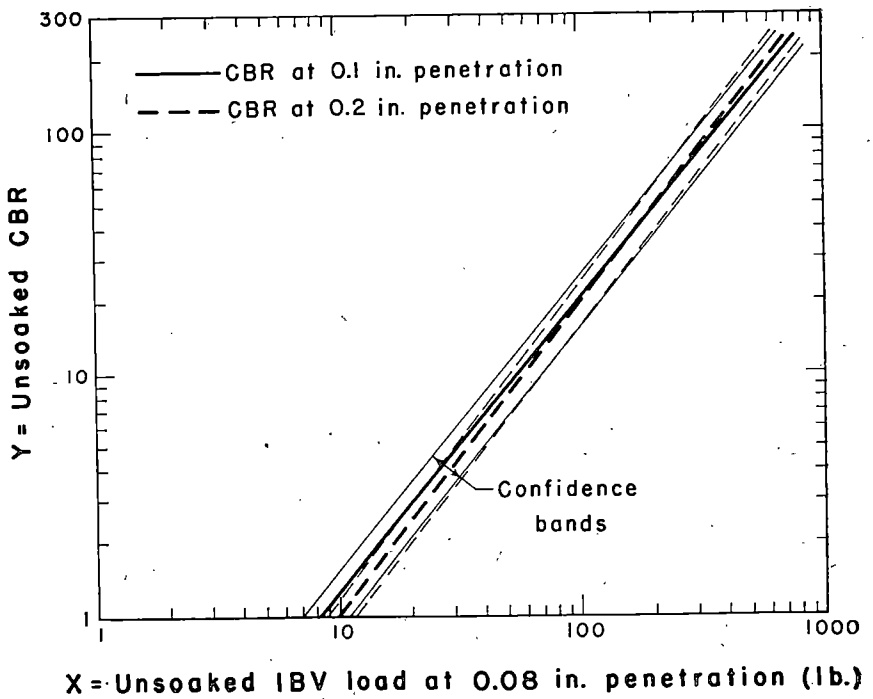

Fig. 2. Prediction of unsoaked CBR of soils with less than 50% sand.

Soaked CBR at 0.1 inch penetration
Under 50% sand $Y = 1.6554X - 2.2088$
50% to 80% sand $Y = 1.3467X - 1.1532$

Soaked CBR at 0.2 inch penetration
Under 50% sand $Y = 1.4110X - 1.6899$
50% to 80% sand $Y = 1.4293X - 1.2354$

In each of these equations Y is the logarithm of the desired CBR, and X is the logarithm of the IBV at the proper penetration, 0.08 inch for unsoaked specimens and 0.10 inch for soaked specimens. These equations are plotted on a log-log scale in figures 2, 3, 4 and 5 to facilitate prediction of the CBR.

Also shown in these figures are the confidence intervals which accompany the prediction curves. These confidence intervals were determined from equation 2 in Appendix B so that ninety-five percent of the points plotted would lie within the intervals.

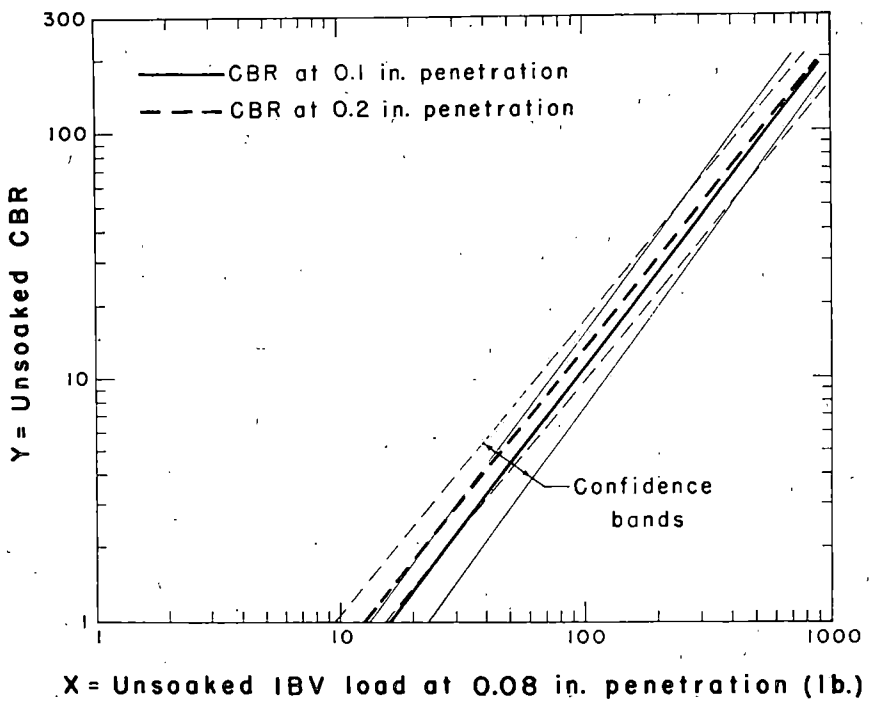

Fig. 3. Prediction of unsoaked CBR of soils with 50% to 80% sand.

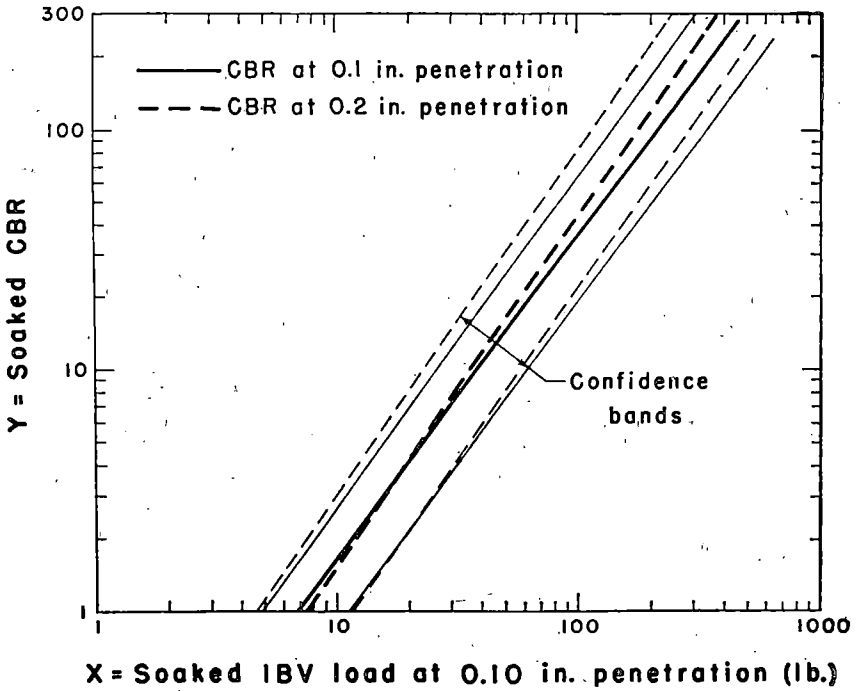

Fig. 4. Prediction of soaked CBR of soils with less than 50% sand.

The widths of the confidence intervals were in all cases reduced from those for previous correlations which did not separate the soils into textural groups[2]. The amount of the reduction ranges from 16.8 percent to 72.8 percent of the previous values.

SUMMARY

For predicting the CBR of a soil or soil-aggregate mixture it is necessary to determine the textural class group of the material and the IBV at 0.08 inch penetration for unsoaked specimens and 0.10 inch penetration for soaked specimens. The proper graph is then selected. The graph is entered at the IBV of the soil which should be the average of three determinations, and the CBR corresponding to 0.1 or 0.2 inch penetration is read directly.

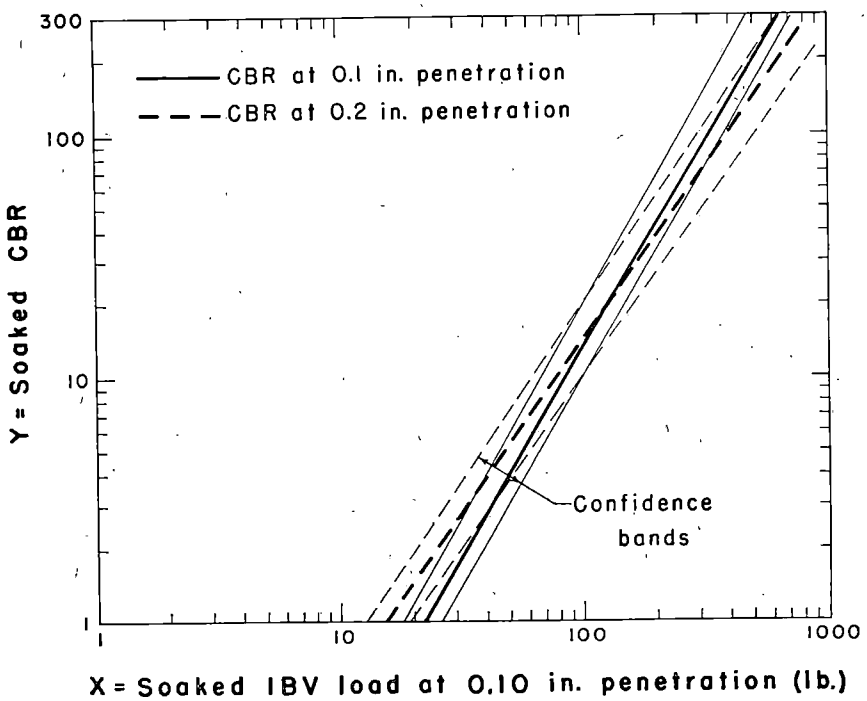

Fig. 5. Prediction of soaked CBR of soils with 50% to 80% sand.

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APPENDIX A

METHOD FOR THE IOWA BEARING VALUE TEST

Scope

This method covers a procedure for determining the supporting strength of soils and soil-aggregate mixtures which pass the No. 10 (2 mm) sieve 100 percent and do not contain more than 80 percent of sand-size material.

Apparatus (figure 6)

Mold—A cylindrical metal mold having an internal diameter of 2.0 ± 0.001 inches and a height of 5.0 ± 0.005 inches is used. The mold is provided with a detachable collar of approximately 2 inches in height.

Base—The base is cylindrical with a diameter of $1\frac{15}{16}$ inches and a height of 3.0 ± 0.001 inches.

Temporary Supports—Temporary supports, approximately 2 inches in height, hold the mold above the bottom of the base until after the first blow with the hammer.

Frame—A frame of two steel rods, a base plate, and a cross-member having a semi-circular notch guides the hammer during compaction. The frame has a lever for forcing the mold to the bottom of the base after molding.

Hammers—Two metal hammers, one 5 lb. and one 10 lb., drop 12 inches. The 5 lb. hammer is used for standard Proctor density; the 10 lb. one for modified AASHO.

Penetration Rod—A steel penetration rod $\frac{5}{8}$ inch in diameter indicates depth of penetration with a dial.

Guiding Device—A metal device fits over the mold during testing and maintains the penetration rod in a vertical position.

Testing Machine—A machine capable of constant movement of the testing head continuously indicates the load.

Soaking Frame—A frame with perforated base holds the mold during immersion.

Annular Weight—A weight with perforated base and spacer ring weighs 550 grams.

Balance—A balance with a capacity of 1000 g sensitive to 0.1 g is required.

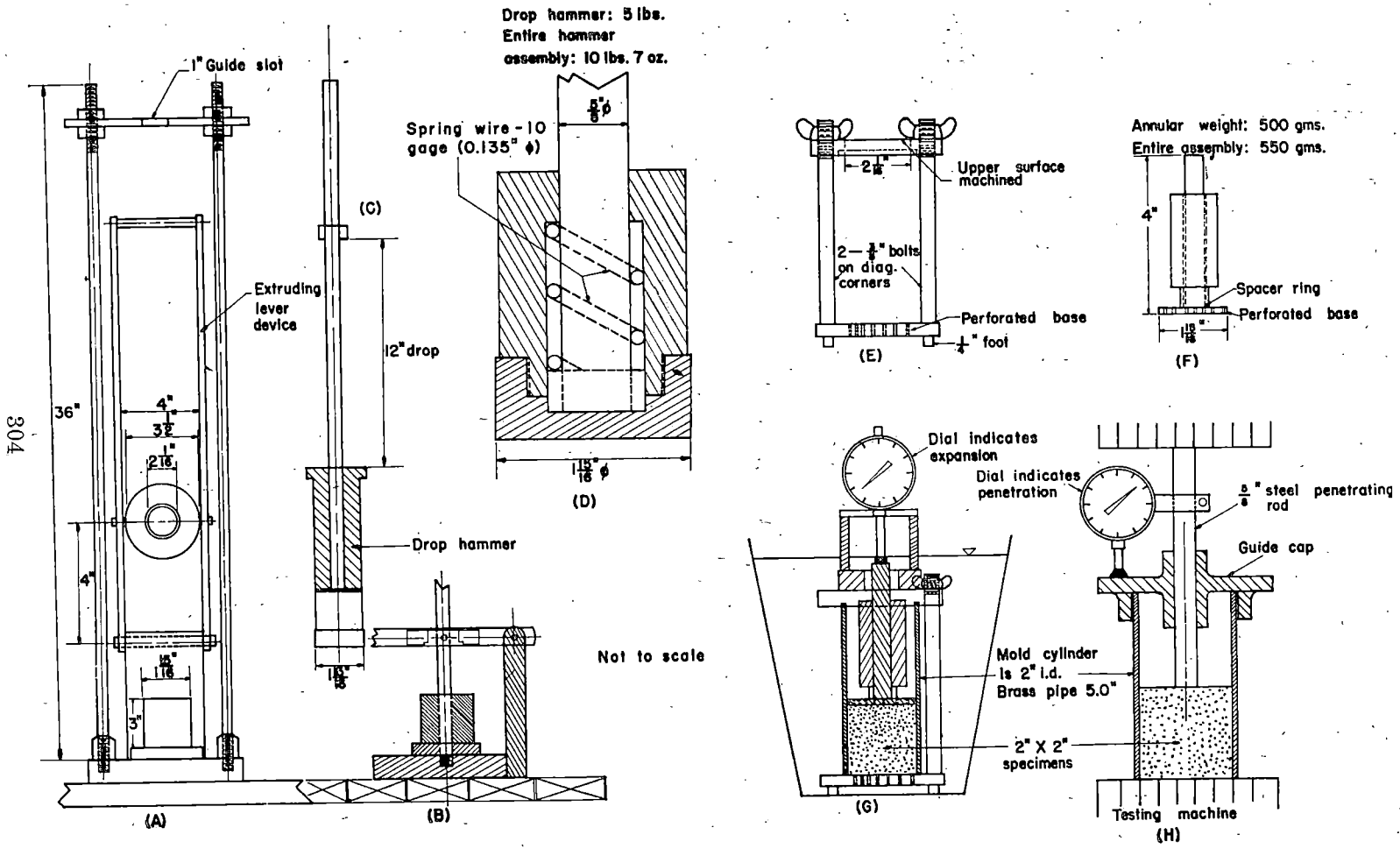


Fig. 6. IBV test apparatus. (A) molding frame, front view; (B) molding frame, side view; (C) drop hammer; (D) drop hammer head; (E) soaking frame; (F) annular weight assembly; (G) specimen during soaking with expansion dial in place; (H) specimen during penetration test.

Straightedge—A rigid steel straightedge with one beveled edge is required.

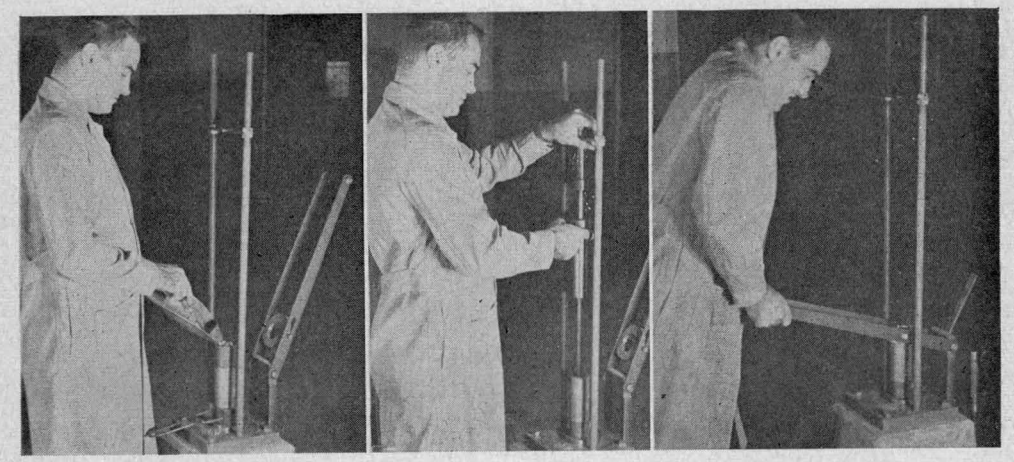
Mixing Tools—Miscellaneous tools include a mixing pan, spoon, trowel, spatula, or a suitable mechanical mixer for mixing the soil thoroughly with water.

Container—A suitable container is used for immersion of specimens.

Sample

Prepare the sample by breaking up soil aggregations to pass the No. 10 (2 mm) sieve in such a manner as to avoid reducing the natural size of the individual particles.

Weigh a representative sample large enough to form three 2 inch diameter by 2 inch high specimens from the soil prepared as in paragraph (a) above.



Pouring soil into the mold. Compacting the specimen. Extruding that part of the specimen in excess of 2 inches.

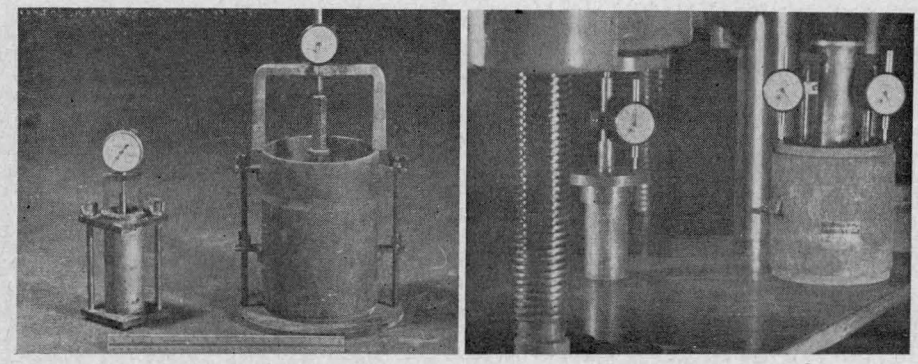
Fig. 7. Preparation of the IBV specimen.

Procedure

Add the required amount of water to the soil and mix thoroughly.

Form a specimen in one layer by compacting the soil in the mold, which has the collar attached and is supported on the temporary supports (figure 7). When compacting to standard Proctor density use the 5 lb. hammer and compact the specimen with five blows on each end. When compacting to modified AASHO density use the 10 lb. hammer and compact the specimen with ten blows on each end. Remove the temporary supports after the first blow with the hammer. After compaction force the specimen to one end of the mold by pushing the mold down around the base with the lever. With the straightedge strike off the excess portion of the specimen.

If the specimen is to be soaked, place the mold in the soaking frame with the specimen at the bottom, place the annular weight assembly on the specimen, and immerse the entire assembly in the soaking container for a period of 48 hours (figure 8). Before testing permit the specimen to drain for five minutes.



IBV and CBR specimens prepared for immersion with expansion reading devices in place.

Penetration test of IBV specimen; the CBR specimen is ready for testing.

Fig. 8. Comparison of IBV and CBR specimens.

To test the specimen place the guiding device over the mold and insert the penetration rod, being careful not to disturb the surface of the specimen. Apply the load so that the rate of penetration is 0.05 inch per minute. Record the load at increments of 0.02 inch up to a penetration of 0.20 inch. The Iowa Bearing Value of the material at any penetration is equal to the mean of the loads on three specimens at that penetration.

Report

The report shall include the following:

Moisture content at which specimen was molded;

Density of specimen;

Absorption data;

Load at each increment of penetration.

APPENDIX B

STATISTICAL ANALYSIS

The purpose of the statistical analysis in this work was to determine the IBV-CBR relationship for the desired CBR penetrations, soaking periods, and sand contents of the samples, and also to determine 95 percent confidence intervals for prediction of the CBR from the IBV.

As pointed out by both Lafleur[5] and Arpacioglu[2] the range of IBV penetrations from which the relationship can be determined is very narrow.

Since the linear relationship between the logarithms of the CBR and IBV values had been established, the relationship could be expressed by the equation:

$$Y = \alpha X + \beta$$

where X and Y are the logarithms of the IBV and CBR values respectively.

The method used to determine the parameters α and β was chosen because this method gave the most satisfactory results of any of the four methods used in the previous correlations. In this method the parameters were estimated from the following equations:

$$\hat{\alpha} = \frac{S_{yyB} - S_{yyW}}{S_{xyB}} \qquad \hat{\beta} = \overline{Y}\,\ldots - \overline{\alpha x}\,\ldots \tag{1}$$

where

$$S_{yyB} = \frac{r_2}{n-1} \sum_{i=1}^{n} \overline{(y_i} - \overline{y}\,\ldots)^2$$

$$S_{yyW} = \sigma^2{}_f = \frac{1}{n(r_2 - 1)} \sum_{i=1}^{1} \sum_{j=1}^{r_2} (y_{ij} - \overline{y_i}\,.)^2$$

$$S_{xyB} = \frac{r_2}{n-1} \sum_{i=1}^{n} (\overline{x_i}\,. - \overline{x}\,\ldots)\ (\overline{y_i}\,. - \overline{y}\,\ldots)$$

and r_1 and r_2 are the number of IBV and CBR readings, respectively, taken at each treatment; and n is the number of tests.

The limits of the confidence intervals were estimated from the equation: in which the number 1.96 is taken from the normal integral.

$$y = \hat{\alpha}x + \hat{\beta} \pm 1.96 \sqrt{\left[\frac{\hat{\sigma}_f^2}{r_2} + \frac{\hat{\alpha}^2\hat{\sigma}_e^2}{r_1}\right]\left[\frac{n+1}{n} + \frac{(x - \bar{x}..)^2 + \frac{\hat{\sigma}_e^2}{nr_1}}{(\bar{x}_i. - \bar{x}..)^2 - (n-1)\frac{\alpha^2_e}{r_1}}\right]} \qquad (2)$$

The accuracy of prediction of the CBR may be still further increased by increasing the number of IBV tests used to determine the IBV of a material. All of the work to date has been done by using the mean of three IBV trials. If the number of trials were increased the width of the confidence intervals would be decreased by the square root of the ratio of the usual number of trials r_1 to the new number of trials r_1'.